FINAL

WORK PLAN

For

BASEWIDE GROUNDWATER MONITORING PROGRAM ALAMEDA POINT, ALAMEDA, CALIFORNIA

August 2015

Prepared for

Naval Facilities Engineering Command Southwest
San Diego, California

Prepared Under

Contract N62473-12-D-2012
Task Order 0096

DCN: MMEC-2012-0096-0003

Prepared by

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ACRONYMS AND ABBREVIATIONS

Alameda Point	Former Naval Air Station Alameda
amsl	above mean sea level
APP	Accident Prevention Plan
BCT	BRAC Cleanup Team
BGMP	Basewide Groundwater Monitoring Program
bgs	below ground surface
BMP	best management practice
BRAC	Base Realignment and Closure
BSU	Bay Sediment Unit
BV	background value
CEG	Certified Engineering Geologist
CERCLA	Comprehensive Environmental Restoration, Compensation, and Liability Act
CPR	Contractor Production Report
CQC	Contractor Quality Control
CSO	(BRAC) Caretaker Site Office
DCA	1,1-dichloroethane
DCB	1,3-dichlorobenzene
DCE	1,1-dichloroethene
DFOW	Definable Feature of Work
DoD	Department of Defense
DOT	Department of Transportation
DTSC	California Department of Toxic Substances Control
EDWP	Exception to Drinking Water Policy
EPP	Environmental Protection Plan
FWBZ	first water bearing zone
HAZWOPER	Hazardous Waste Operations and Emergency Response
HSZ	hydrostratigraphic zone
IC	Institutional Control
ICTC	Institutional Control Termination Criterion
IDW	investigation-derived waste
IR	Installation Restoration
ITSI	Innovative Technical Solutions, Inc.
LUC	Land Use Control
MEC	munitions and explosives of concern

ACRONYMS AND ABBREVIATIONS (CONTINUED)

mg/L	Milligrams per liter
MGP	manufactured gas plant
mL/min	milliliters per minute
MMEC Group	Multimedia Environmental Compliance Group
msl	mean sea level
NAVFAC SW	Naval Facilities Engineering Command Southwest
NFA	No Further Action
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PCE	tetrachloroethene
PE	Professional Engineer
PG	Professional Geologist
PM	Project Manager
PMO	Program Management Office
POC	Point of Contact
PPE	personal protective equipment
PVC	polyvinyl chloride
QA	quality assurance
QC	quality control
RG	remedial goal
ROD	Record of Decision
ROICC	Resident Officer in Charge of Construction
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SES-TECH	SES-TECH Remediation Services
Shaw	Shaw Environmental, Inc.
SSHP	Site Safety and Health Plan
SVOC	semivolatile organic compound
SWBZ	second water bearing zone
TCE	trichloroethene
TDS	total dissolved solids
U.S. EPA	United States Environmental Protection Agency
USA	Underground Service Alert
USACE	United States Army Corps of Engineers

ACRONYMS AND ABBREVIATIONS (CONTINUED)

VOC	volatile organic compound
Water Board	California Regional Water Quality Control Board – San Francisco Bay Region
WBZ	shallow water bearing zone
WMP	Waste Management Plan

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1 INTRODUCTION

Naval Facilities Engineering Command Southwest (NAVFAC SW) has retained the Multimedia Environmental Compliance Group (MMEC Group), a joint venture comprising Amec Foster Wheeler Environment & Infrastructure, Inc. KMEA Inc., under Contract No. N62473-12-D-2012, Task Order No. 0096, to perform the Basewide Groundwater Monitoring Program (BGMP) at former Naval Air Station Alameda (Alameda Point), Alameda, California.

This Work Plan includes the following project plans:

- A Sampling and Analysis Plan (SAP) to provide guidance on sampling, analysis, and quality assurance (QA) during the BGMP (United States Environmental Protection Agency [U.S. EPA], 2005).
- A Waste Management Plan (WMP) that describes investigation-derived waste (IDW) generated by the contractor and the method for its disposal.
- An Environmental Protection Plan (EPP) that identifies known or potential environmental issues that may be encountered while conducting the BGMP activities and presents mitigation measures and best management practices (BMPs) that will be implemented.

This Work Plan describes the Alameda Point site and provides background information, presents the project objectives, and outlines the plan and schedule for completing the project in accordance with the NAVFAC SW Scope of Work.

1.1 OBJECTIVE

The primary objective of the BGMP is to monitor groundwater and soil gas conditions at Alameda Point and to provide supplemental data to support proposed and ongoing Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) actions, including natural attenuation, at the various Installation Restoration (IR) and Operable Unit (OU) sites. This continued monitoring provides data to support remedial actions in accordance with Records of Decision (RODs) for Alameda Point. In addition to soil gas and groundwater data collection, optimization of the BGMP and decommissioning of identified wells will be performed under this project.

1.2 REGULATORY FRAMEWORK

Alameda Point is a National Priorities List (NPL) facility. The Navy, as lead agency, has entered into a Federal Facilities Agreement with the following regulatory agencies:

- U.S. EPA, the lead federal regulatory agency
- California Department of Toxic Substances Control (DTSC), the lead state regulatory agency
- Regional Water Quality Control Board–San Francisco Bay Region (Water Board), the state regulatory support agency

1.3 WORK PLAN ORGANIZATION

This Work Plan was prepared in accordance with applicable Navy Environmental Restoration Program guidance and is organized as follows:

- **Section 1**, **Introduction** – scope of work, project objectives, identification of the regulatory agencies and regulatory requirements, and outline of the Work Plan.
- **Section 2**, **Site Description –** history and physical description of Alameda Point.
- **Section 3, BGMP Design and Approach –** summary of history, design, and technical approach for conducting the BGMP.
- **Section 4, Field Implementation** – field activities that will be conducted as part of BGMP.
- **Section 5, Contractor Quality Control Plan –** the quality control plan that will be implemented by the contractor for construction-related activities as part of this project.
- **Section 6, Reporting, Scheduling, and Optimization** – the overall project schedule and plans to report the results of the BGMP.
- **Section 7, References** – documents used to prepare this Work Plan.

Appendices to this Work Plan for the Alameda BGMP are as follows:

- **Appendix A** – Sampling and Analysis Plan, which describes the data quality objectives, the analytical methods, and the laboratory analytical quality standards to support adequate data collection.
- **Appendix B** – Waste Management Plan, which describes the contractual, legal, and risk management requirements in the generation, storage, sampling and analysis, waste-classification, transportation, treatment, and ultimate disposal of waste generated during field activities.
- **Appendix C** – Environmental Protection Plan, which describes best management practices to be followed during field work to ensure protection of the environment.

2 SITE DESCRIPTION

This section presents site background information and a description of the topography, geology, and hydrogeologic setting of the site.

2.1 TOPOGRAPHY

Based on groundwater well survey elevations, the surface elevation at Alameda Point is approximately 7 to 17 feet above mean sea level (amsl). The site is relatively flat, mostly developed, and paved, with some areas having been left unpaved.

2.2 GEOLOGY

Alameda Point occupies a depression between two uplifted areas: the Berkeley Hills to the east and the Santa Cruz Mountains on the San Francisco Peninsula to the west. The depression and uplifted areas are formed by two subparallel, active faults: the San Andreas and the Hayward Faults. Alameda Point and the surrounding San Francisco Bay are underlain by 400 to 500 feet of unconsolidated sediments that overlay the metamorphosed sandstone, siltstone, shale, greywacke, and igneous bedrock, which forms the Franciscan Formation (Bechtel, 2008).

Surface and near-surface soil at Alameda Point consists of artificial fill emplaced during historical filling of the tidal marshlands and the subtidal area of San Francisco Bay during site development. The fill material consists of sediments that were dredged from the tidal flats of San Francisco Bay and Oakland Inner Harbor and is characterized by sands, clays, and silts (Bechtel, 2008). The unconsolidated sediments that lie beneath the artificial fill consist of the following five units, from top to bottom: (1) the Bay Sediment Unit (BSU), (2) the Merritt Sand Formation, (3) the upper unit of the San Antonio Formation, (4) the lower unit of the San Antonio Formation (Yerba Buena Mud), and (5) the Alameda Formation.

2.3 HYDROGEOLOGY

Groundwater is present at Alameda Point at approximately 1 to 8 feet below ground surface (bgs) in the artificial fill. From top to bottom, the following five distinct hydrostratigraphic zones (HSZ) have been identified at Alameda Point (although not all occur at all locations):

- First Water Bearing Zone (FWBZ) (Artificial Fill)
- Semi-confining aquitard (Bay Sediment Unit [BSU]/Young Bay Mud)
- Second Water Bearing Zone (SWBZ) (Posey, Merritt Sand, and upper San Antonio Members)
- Yerba Buena Mud Aquitard, a regional aquitard
- Alameda Aquifer

All units are members of the Alameda Formation, except for the artificial fill.

The BSU, where present, acts as an aquitard between the FWBZ and the SWBZ and is approximately 12 to 35 feet thick, although the BSU pinches out to the southeast at the edge of the historical Alameda Island. The semi-confined SWBZ is composed of the lower portion of the Posey Member, the Merritt Sand Member, and the upper portion of the San Antonio Member and has a maximum thickness of 88 feet. The Yerba Buena Mud Aquitard acts as the regional aquitard (Innovative Technical Solutions, Inc. [ITSI]) 2009; Navy, 2012). In the southeastern part

of Alameda Point (OU-2A and OU-2B, and IR Sites 27 and 35), the BSU is not present because of the past depositional environment. Therefore, the artificial fill and the Posey, Merritt Sand, and the upper portion of the San Antonio Members together make up the unconfined aquifer.

For the purposes of the BGMP, in the areas where the BSU is present, the aquifers are referred to as the FWBZ and SWBZ. In areas where the BSU is absent or not laterally continuous, the unconfined aquifer is referred to as the Shallow Water-Bearing Zone (WBZ) (approximately equivalent to the FWBZ) and the Deep WBZ (approximately equivalent to the SWBZ); however, the Shallow WBZ can be subdivided into a shallow portion and a deeper portion dependent on interpretations of the local hydrogeology at each separate OU or IR site. For example, at OU-2B the feasibility study states that the unconfined/semi-confined WBZ extends to at least 70 feet bgs (SES-TECH, 2015). Therefore, the Shallow WBZ at OU-2B includes wells screened to a depth of 70 feet bgs.

Groundwater in most of the FWBZ and the Shallow WBZ in the western half of Alameda Point is slightly saline. Groundwater at Alameda Point generally flows from the middle of Alameda Point outward toward San Francisco Bay, the Oakland Inner Harbor, and Seaplane Lagoon. Groundwater flow directions may vary throughout the year because of seasonal changes in precipitation and tidal changes (ITSI, 2009).

2.3.1 Beneficial Use Determination

This summary of the beneficial use determination for Alameda Point is based on information provided in the final Work Plan for the BGMP from 2004 (Shaw Environmental, Inc., 2004a). Additional details are provided in that Work Plan and in the documents cited therein. The beneficial use study report (Tetra Tech EM Inc., 2000) classifies groundwater across Alameda Point on the basis of the Guidelines of Groundwater Classification under the U.S. EPA Groundwater Protection Strategy (U.S. EPA, 1988). Criteria used to classify groundwater include total dissolved solids (TDS) concentration and minimum well yield (ITSI, 2009). The classifications are as follows (U.S. EPA, 1986):

- Class I: An irreplaceable source of drinking water or ecologically vital
- Class II: Current or potential source of drinking water and with other beneficial use
- Class III: Not a potential source of drinking water and with limited beneficial use

In the western portion of Alameda Point, the FWBZ beneath IR Sites 1 and 32 is Class II groundwater based on federal TDS concentration and yield criteria. Groundwater beneath IR Site 2 is Class III groundwater. The SWBZ within the western area of Alameda Point is designated as Class III groundwater because TDS concentrations exceed 10,000 milligrams per liter (mg/L). Based on these factors and planned property redevelopment, the Alameda Point Base Realignment and Closure (BRAC) Cleanup Team (BCT) concluded that the groundwater beneath IR Sites 1, 2, and 32 is unlikely to be used as a potential future drinking water source (ITSI, 2009).

The FWBZ in the central portion of Alameda Point beneath IR Site 5 is considered Class II groundwater based on federal TDS concentration and yield criteria. Based on other mitigating factors and property reuse, the BCT concluded that groundwater beneath IR Site 5 is not likely to be used as a drinking water source (ITSI, 2009).

Groundwater in the southeastern portion of Alameda Point has been de-designated from municipal use and is not subject to domestic groundwater use standards, including all of OU-2A

(IR Sites 9, 13, 19, 22, and 23) and OU-2B (IR Sites 3, 4, 11, and 21), IR Site 27, and a portion of IR Site 35 that abuts IR Site 21 (Navy BRAC PMO, 2012). A groundwater exception to drinking water policy (EDWP) was determined by the Water Board on September 13, 2012 for shallow groundwater (Water Board, 2012). The groundwater was de-designated for the following reasons: (1) TDS values exceeding 10,000 mg/L, (2) the shallow groundwater's communication with San Francisco Bay, (3) an absence or lack of lateral continuity of the clay aquitard (Young Bay Mud) to create two separate aquifers, (4) state well construction requirements (inability to meet them), and (5) current and anticipated future use of groundwater at Alameda Point. As a result of the 2012 groundwater EDWP, the drinking water ingestion exposure pathway no longer exists, and the remediation goals (RG) for shallow groundwater have been revised from those protective of drinking water standards to those protective of potential vapor intrusion risk to a commercial/industrial worker. Therefore, the ROD remedial action objective for the southeastern portion of Alameda Point has been modified to, "protect the beneficial use of the aquifer…with the exception of the municipal and domestic water supply beneficial use for shallow groundwater from ground surface to 79 ft, or the top of the Yerba Buena mud" (MMEC Group, 2014).

Finally, the Water Board has de-designated groundwater as a drinking water source at Alameda Point beneath the entire western portion and beneath part of the central portion (west of Saratoga Street) because of the factors mentioned above in accordance with (1) the East Bay Plain Groundwater Basin Beneficial Use Evaluation Report (Water Board, 1999); (2) the Proposed Groundwater Amendments to the Water Quality Control Plan (Water Board, 2000a); and (3) Board Resolution 00-024 (Water Board, 2000b) tidal influence studies, which are discussed in more detail below.

2.4 SITE HISTORY

Alameda Point is located on the western end of the island of Alameda. The site encompasses 2,634 acres, which consists of approximately 1,600 acres of dry land and 1,000 acres of submerged tideland. Alameda Point has been owned and operated by the Department of Defense (DoD) since 1930 and has served as a naval aircraft maintenance, repair, and refit center. It was also used as a base of operation for naval surface craft (Shaw, 2004a). Upon closure in April 1997, the site was renamed Alameda Point in a reorientation of the facility toward civilian use. In July 1999, Alameda Point was identified as an NPL site and the Navy is currently conducting environmental activities in accordance with CERCLA.

Before 1850, the site was a much smaller peninsula consisting of dry land and marshland. The "island" of Alameda was created in 1902 when dredging of the San Leandro Channel was completed (Bechtel, 2008). As a result of fill projects conducted between 1871 and 1961, Alameda Island is now three times its original size. Only the eastern portion of Alameda Point consisted of original, dry land. Most of the site is located on fill up to 40 feet thick (Bechtel, 2008). From the late 1800s until the 1920s, two manufactured gas plants (MGP) and an oil refinery located near present-day Alameda Point apparently discharged petroleum products and other wastes to adjacent marshlands. These wastes then spread through tidal action over the marsh surface and tidal flats. As the marshlands and subtidal areas were filled, the discharged petroleum products became entrapped in the subsurface, creating what is now referred to as the Marsh Crust and subtidal area (Bechtel, 2008). The Marsh Crust and subtidal area material consists of a thin (up to 6-inch-thick) layer of hydrocarbon-contaminated sediment indicative of coal-based MGP-derived waste.

Wastes historically disposed of at the site included solvents, waste oil, paints, scrap metal, garbage, radiological material, aircraft engines, and possibly munitions and explosives of concern (MEC). In addition, it is estimated that 365,000 gallons of aviation fuel have leaked from underground fuel tanks (ITSI, 2009). Numerous plating and painting shops are also being investigated to determine the extent of contamination resulting from past spills and other disposal practices.

The Alameda Point BGMP provides groundwater and soil gas analytical data to be used, in conjunction with other information generated by ongoing site investigation and remedial actions, to define the nature and extent of contamination in groundwater at Alameda Point (ITSI, 2009). A number of individual or commingled groundwater plumes have been identified at Alameda Point. The groundwater plumes generally consist of chlorinated constituents, petroleum hydrocarbons, and other fuel-related compounds, metals, or a combination of these contaminants (ITSI, 2009). To monitor these plumes, groundwater samples are routinely collected from selected groundwater monitoring wells and are analyzed for site-specific analytes.

Various IR sites have been defined at Alameda Point, some of which are subject to BGMP activities. In some cases, multiple IR sites were grouped into various OUs. Groundwater sampling and/or landfill gas monitoring was conducted following methods and procedures described in previous SAP documents (Shaw, 2004b, c; ITSI, 2004). However, since that time, conditions at the various IR sites have changed, or in some cases, IR sites have been removed from the BGMP. The history and current state of the BGMP are discussed in greater detail later in this document.

3 BASEWIDE GROUNDWATER MONITORING PROGRAM DESIGN AND APPROACH

The following sections provide a summary of the BGMP, the basis for comparison criteria used at each OU/IR site, a summary of changes to the BGMP, an optimization summary, and the problem statement for the BGMP.

3.1 BASEWIDE GROUNDWATER MONITORING PROGRAM SUMMARY

The primary purpose of the BGMP is to monitor groundwater and soil gas conditions at Alameda Point and to provide supplemental data to support proposed and ongoing CERCLA actions (including natural attenuation). Several individual and commingled groundwater plumes have been identified at Alameda Point. The groundwater plumes generally consist of chlorinated hydrocarbons, petroleum hydrocarbons, other fuel-related compounds, metals, radionuclides, or a combination of these contaminants (SES TECH, 2015). To monitor these plumes, groundwater samples are routinely collected from selected monitoring wells and analyzed for site-specific chemicals of interest. The BGMP provides for monitoring at Alameda Point and does not provide for detailed or comprehensive interpretations of site conditions in the various OUs and IR sites at Alameda Point. Site-specific information sufficient to make CERCLA decisions will be discussed and reported in IR site-specific documents. The site-specific documents can rely on BGMP data, but the BGMP report does not serve as a Remedial Action Closure Report for a specific site. Site-specific CERCLA evaluations are being executed through documentation generated under OU-specific contracts other than the BGMP task order. A summary of the current status of each IR site and a reference to site-specific documents which contain detailed descriptions of each site's conceptual site model is provided in Worksheet #10 of the SAP (Appendix A).

3.2 COMPARISON CRITERIA

Results for groundwater samples collected as part of the BGMP are compared with criteria established specifically for each IR site. RGs and comparison criteria for each IR site is presented further in Worksheet #17 of the SAP (Appendix A). RGs and comparison criteria, such as those published in the ROD or Proposed Plans for the IR sites, are as follows (not all of which still are being monitored under the BGMP):

- **IR Site 1:** The RGs for IR Site 1 surface water are based on occupational and recreational exposure pathways for inhalation of volatile organic compounds (VOCs) in ambient air, including benzene, 1,1-dichloroethene (DCE), trichloroethene (TCE). The RG for IR Site 1 groundwater exposure is for vinyl chloride (Navy BRAC PMO, 2009).
- **IR Sites 3, 4, 11, and 21:** The Proposed Plan presents proposed RGs for risk associated with inhalation of vapors from groundwater for TCE and vinyl chloride and for surface water discharge to the Seaplane Lagoon for TCE (Navy BRAC PMO, 2013). As of September 2012, shallow groundwater at these IR sites has been exempted as a drinking water source (Water Board, 2012).
- **IR Site 5:** The ROD presents RGs for 1,1-dichloroethane (DCA), TCE, and vinyl chloride in shallow groundwater. For deep FWBZ and SWBZ groundwater, RGs are proposed for vinyl chloride and total VOCs (Navy BRAC PMO, 2014).

- **IR Site 13:** The commercial vapor intrusion criteria are the IR Site 13 remedial action RGs, and the residential vapor intrusion values are the Institutional Control (IC) termination for residential reuse restrictions for benzene and ethylbenzene (Navy BRAC PMO, 2012).
- **IR Site 16:** The exposure pathway is assumed to be commercial/industrial use vapor inhalation from groundwater. The RGs for IR Site 16 are for 1,3-dichlorobenzene (DCB), 1,4-DCB, cis-1,2-DCE; tetrachloroethene (PCE), TCE, and vinyl chloride in groundwater (Navy BRAC PMO, 2007a).
- **IR Site 26:** The RGs for IR Site 26 are based on a residential indoor air vapor inhalation pathway from groundwater and include cis-1,2-DCE, TCE, and vinyl chloride in groundwater (Navy BRAC PMO, 2006).
- **IR Site 27:** The RGs for IR Site 27 are for 1,1-DCA, cis-1,2-DCE, PCE, trans-1,2-DCE, TCE, and vinyl chloride in groundwater (Navy BRAC PMO, 2008).
- **IR Site 28:** The RGs for IR Site 28 are for arsenic and copper in groundwater. The exposure pathway is considered to be domestic (residential) use of groundwater (Navy BRAC PMO, 2007b).
- **IR Sites 1 and 32:** Because of historical high values for radionuclides detected in groundwater at IR Sites 1 and 32, radionuclides are included in the BGMP for these sites (Navy BRAC PMO, 2009 and 2010).
- **IR Site 2:** The comparison criteria set forth in the ROD for IR Site 2 are groundwater background values (BVs) for arsenic, copper, and nickel (Navy BRAC PMO, 2010). In accordance with the Final Remedial Action Work Plan, Revision 1, (Tetra Tech EC, 2013), groundwater samples collected since completion of the remedial action are also analyzed for pesticides and the semivolatile organic compounds (SVOCs), including bis[2-ethylhexyl]phthalate, in addition to the original ROD-specified dissolved metals.
- **IR Site 14:** The ROD for IR Site 14 sets a Land Use Control (LUC) and defines an Institutional Control Termination Criterion (ICTC) for vinyl chloride in groundwater (Navy BRAC PMO, 2007c). The indoor air risk estimate for residential receptors identified vinyl chloride as the only groundwater risk driver.

BVs are described in the final remedial investigation report for OU-1 and IR Sites 6, 7, 8, and 16 (Tetra Tech EM Inc., 2004) approved by the BCT. BVs apply to all groundwater at Alameda Point.

The RG, ICTC, and LUC termination comparisons for each IR site are defined in Table 17-1 of the SAP.

3.3 CHANGES TO BASEWIDE GROUNDWATER MONITORING PROGRAM

A number of changes to the BGMP were proposed after Navy and regulatory agency review of the draft 2010 Annual Report. Beginning with the fall 2011 event, some of the 2010 BGMP recommendations were implemented by the Navy, with a few additional changes implemented starting with the spring 2012 event. Since then, recommendations for BGMP optimization made in successive annual reports have been accepted and implemented, beginning in the fall of the following annual monitoring period and ending with the Final 2013 annual BGMP monitoring

report (SES-TECH, 2014). Recommended changes to the BGMP from the final 2013 report were implemented during the 2013–2014 sampling period reported in the 2014 annual report.

The Navy has terminated preparation of groundwater elevation contour maps beginning with the 2014 annual report. For future reporting of the BGMP, the groundwater elevation measurements will be tabulated and reported for each IR site, but no groundwater contour maps will be prepared and reported for each IR site. This is because this information has been presented in previous reports and has not substantially changed. Future figures showing individual IR sites in the annual reports will have an arrow indicating the direction of groundwater flow that has been reported and verified in previous years.

Historically, monitoring wells have been added or removed from the BGMP on the basis of specific IR site data needs and to address regulatory agency suggestions. Monitoring wells sampled also have been adjusted on the basis of review of contaminant trends and the preceding year's data. IR sites may also be added or removed from the BGMP depending on whether a project is ongoing at that site under a separate contract. This approach will continue throughout the duration of the BGMP conducted under this Work Plan and SAP. A summary of changes to the BGMP since from 2007 to 2014 is presented in Table 3-1.

Table 3-1.
BGMP Changes Summary

OU No.	IR Site No.	Changes	Implementation Schedule
OU-1	IR Site 6	Discontinue sampling for TPH, SVOCs and metals	Summer 2008
		Discontinue sampling at IR Site 6	Summer 2009
		Discontinue groundwater elevation measurements at IR Site 6	Fall 2009
		Add sampling at IR Site 6 for Fall 2012 only	Fall 2012
		Add sampling at IR Site 6 for Fall 2013 only	Fall 2013
	IR Site 7	Discontinue sampling for NAPs	Spring 2008
		Discontinue sampling at IR Site 7	Summer 2008
		Discontinue groundwater elevation measurement at IR Site 7	Fall 2009
	IR Site 8	Discontinue sampling at IR Site 8	Spring 2008
		Discontinue groundwater elevation measurement at IR Site 8	Fall 2009
	IR Site 14	Discontinue sampling at IR Site 14	Spring 2008
		Discontinue groundwater elevation measurement at IR Site 14	Fall 2009
		Add sampling at IR Site 14	Fall 2010
		Change sampling frequency at M14-06, M14-09S, M14-09D, M14-15, M14-17, and M14-20D from quarterly to semiannually	Fall 2014
		Discontinue sampling at wells M14-14, M14-16, and M14-18	Fall 2014
	IR Site 15	Discontinue groundwater elevation measurement at IR Site 15	Fall 2009
	IR Site 16	Discontinue sampling for PCBs	Summer 2008
		Discontinue sampling at IR Site 16	Summer 2009
		Discontinue groundwater elevation measurement at IR Site 16, except at wells MW16-05 and MWC2-2	Fall 2009
		Add sampling at IR Site 16 for Fall 2012 only	Fall 2012
		Add sampling at IR Site 16 for Fall 2013 only	Fall 2013
		Discontinue groundwater elevation measurement at IR Site 16	Fall 2014

Table 3-1.
BGMP Changes Summary (continued)

OU No.	IR Site No.	Changes	Implementation Schedule
		Add sampling at IR Site 16 for VOCs and WQ parameters for Fall 2014 only	Fall 2014
OU-2A	IR Site 9	Add sampling for 1,4-dioxane at wells MW410-2 and MW410-3	Fall 2007
		Discontinue sampling for NAPs	Spring 2008
		Discontinue sampling at well MW410-1	Fall 2009
		Add sampling at wells S9-TT-MW01, S9-TT-MW02, and S9-TT-MW03	Spring 2010
		Add sampling at wells 9IF-MW01L, 9IF-MW01S, 9IF-MW01U, 9IF-MW02L, 9IF-MW03L, 9IF-MW03U, 9IF-MW04U, 9IF-MW05L, 9IF-MW05U, 9IF-MW06L, 9IF-MW06U, 9IF-MW07L, 9IF-MW07U, 9IF-MW08U, 9IF-MW09L, 9IF-MW10L, 9IF-MW10U, 9IF-MW11U, D09-01, F9SMW03, F9SMW04, F9SMW05, 9EMW01, 9EMW03, 9EMW04, P-9-MWI05, and P-9-MWS01	Fall 2010
		Add TDS analysis to wells D09-01, 9IF-MW09L, 9IF-MW10L, S9-TT-MW01, S9-TT-MW02, S9-TT-MW03, and MW410-3	Fall 2011
		ROD final, no monitoring required; site removed from BGMP	Fall 2012
	IR Site 13	Discontinue sampling for NAPs	Spring 2008
		Discontinue groundwater elevation measurement at well M-IMF-1	Fall 2009
		Add sampling at well S13-TT-MW01	Spring 2010
		Add TDS analysis to well S13-TT-MW01	Fall 2011
		Discontinue sampling at wells M13-06, M13-07, M13-08, M13-09, and S13-TT-MW01	Fall 2012
		Discontinue groundwater elevation measurement at IR Site 13	Fall 2014
		Discontinue sampling at IR Site 13	Fall 2014
	IR Site 19	Add sampling for 1,4-dioxane at well MWD13-4	Fall 2007
		Discontinue sampling for NAPs	Spring 2008
		Add sampling at well S19-TT-MW01	Spring 2010
		NFA; ROD final, no monitoring required; site removed from BGMP	Fall 2012
	IR Site 22	Discontinue sampling for NAPs	Spring 2008
		NFA; ROD final, no monitoring required; site removed from BGMP	Fall 2008
	IR Site 23	Discontinue sampling for natural attenuation parameters	Spring 2008
		Change sampling frequency for VOCs at well MWOR-5 from quarterly to semiannually	Spring 2009
		NFA; ROD final, no monitoring required; site removed from BGMP for soil and groundwater	Fall 2012

Table 3-1.
BGMP Changes Summary (continued)

OU No.	IR Site No.	Changes	Implementation Schedule
OU-2B	IR Site 3	Discontinue sampling for NAPs	Spring 2008
		Add sampling at well S3-TT-MW01	Spring 2010
		Discontinue groundwater elevation measurement at well M03-15 because well has been destroyed	Spring 2011
		Discontinue sampling at wells 398-MW4, M03-07, and MW97-3	Fall 2012
		Discontinue sampling at IR Site 3	Fall 2014
	IR Site 4	Discontinue sampling for NAPs	Spring 2008
		Change sampling frequency for VOCs at well D03-03 from annually to quarterly	Spring 2009
		Add sampling at wells S4-TT-MW01, S4-TT-MW02D, S4-TT-MW02E, S4-TT-MW03C, S4-TT-MW03D, and S4-TT-MW03	Spring 2010
	IR Site 11	Discontinue sampling for NAPs	Spring 2008
		Discontinue sampling for 1,4-dioxane at wells M03-15 and M03-16	Fall 2008
		Add sampling at wells S11-TT-MW01A, S11-TT-MW01B, S11-TT-MW01C, S11-TT-MW02A, S11-TT-MW02B, S11-TT-MW02C, S11-TT-MW03A, S11-TT-MW03B, S11-TT-MW03C, S11-TT-MW04A, S11-TT-MW04B, S11-TT-MW04C, S11-TT-MW05A, S11-TT-MW05B, S11-TT-MW05C, S11-TT-MW06A, S11-TT-MW06B, S11-TT-MW06C, S11-TT-MW01D, S11-TT-MW01E, S11-TT-MW02D, S11-TT-MW02E, S11-TT-MW03D, S11-TT-MW03E, S11-TT-MW04D, S11-TT-MW04E, S11-TT-MW05D, S11-TT-MW05E, S11-TT-MW06D, and S11-TT-MW06E	Spring 2010
		Nested wells S11-TT-MW02A through S11-TT-MW02E and S11-TT-06A through S11-TT-06E were destroyed due to on-site remediation activities	Spring 2011
		Discontinue sampling at wells S11-TT-MW02A, S11-TT-MW02B, S11-TT-MW02C, S11-TT-MW02D, S11-TT-MW02E, S11-TT-MW06A, S11-TT-MW06B, S11-TT-MW06C, S11-TT-MW06D and S11-TT-MW06E	Fall 2012
	IR Site 21	Discontinue sampling for NAPs	Spring 2008
		Change sampling frequency for metals at wells OU2B-MW03, OU2B-MW04, and OU2B-MW05 from quarterly to semiannually	Summer 2008
		Discontinue analysis for dissolved metals	Fall 2013

Table 3-1.
BGMP Changes Summary (continued)

OU No.	IR Site No.	Changes	Implementation Schedule
OU-2C	IR Site 5	Discontinue sampling at well M117-E	Fall 2007
		Add sampling for 1,4-dioxane at wells 2MW8S, M05-04, M05-06, M05-08, M05-09, M05-11, and M05HW-01	Fall 2007
		Add sampling at wells L05-01, L05-02, L05-03, L05-04, L05-05, L05-06, M05-02, M05-05, M05-13, M05-14, M05-15, M05-16, M05-17, M05-18, M05-19, M05-20, and D05-09	Fall 2007
		Discontinue sampling at well D05-01	Spring 2009
		Change sampling frequency for all analytes from quarterly to semiannually	Spring 2009
		Discontinue sampling at well MLS-6	Fall 2009
		Discontinue analysis for dissolved metals and NAPs	Fall 2013
		Discontinue groundwater elevation measurement at IR Site 5	Fall 2014
		Discontinue sampling at IR Site 5 and decommission wells	Fall 2014
	IR Site 10	Add sampling for 1,4-dioxane at wells M10-01 and M10-02	Fall 2007
		Discontinue sampling at wells M10-01 and M10-02	Fall 2012
		Discontinue groundwater elevation measurement	Fall 2013
	IR Site 12	Discontinue sampling at well M12-04	Fall 2007
		Discontinue sampling at well D12-01	Spring 2009
		Discontinue groundwater elevation measurement	Fall 2013
OU-3	IR Sites 1 and 32	Change soil gas sampling frequency for TO-15 from quarterly to semiannually or annually	Spring 2009
		Discontinue groundwater elevation measurement at well M026-E	Fall 2009
		Remove TO-15 analysis for soil gas samples	Spring 2011
		Discontinue sampling at wells M001-A, M001-B, M003-B, M027-B, M027-C, M028-C, and M030-C	Fall 2012
		Discontinue sampling at wells M004-A, M025-C, M031-C and IR32-MW-08	Fall 2012
	IR Site 32	Add sampling at wells IR32-MW-06, IR32-MW-07, and IR32-MW-08	Fall 2010
		Remove TO-15 analysis for soil gas samples	Spring 2011
		Discontinue sampling at IR Site 32 wells M004-A, M025-C, M031-C and IR32-MW-08	Fall 2012
		Change sampling frequency from quarterly to semiannually	Winter 2012
OU-4A	IR Site 2	Discontinue sampling for NAPs	Spring 2008
		Change soil gas sampling frequency for TO-15 from quarterly to semiannually or annually	Spring 2009
		Add sampling at wells M108-A, M009-A, M103-A, and M103-B	Spring 2010
		Add sampling at wells M108-B and DRA-01	Fall 2010
		Remove TO-15 analysis for soil gas samples	Spring 2011
		Discontinue sampling at wells M010-A, M016-A, M018-A, M019-A, M021-A, M022-A and M036-A	Fall 2012

Table 3-1.
BGMP Changes Summary (continued)

OU No.	IR Site No.	Changes	Implementation Schedule
		Replace wells damaged during landfill cover construction, and revise analysis in accordance with RAWP, Revision 1.	Fall 2013
OU-5	IR Sites 25 Group	Discontinue sampling at IR Site 25 Group	Summer 2007
OU-6	IR Site 26	Discontinue groundwater elevation measurement at IR Site 26, except at wells 26SW04 and 26SW05	Fall 2009
		Add sampling at wells 26MW01 through 26MW05, 26MW07 through 26MW09, 26PZ01 through 26PZ03, and BSU-1	Fall 2011
		Discontinue sampling at wells 26MW01, 26MW02, 26MW04, 26MW05, 26MW07, 26MW09, and BSU-1	Fall 2012
	IR Site 27	Discontinue sampling at IR Site 27	Summer 2007
		Discontinue groundwater elevation measurement at IR Site 27, except at wells 27MW07 and 27MW08	Fall 2009
		Add sampling at wells 27MW01, 27MW02, 15MW02, 27EW-05, 27EW-15, 27EW-19, 27EW-20, 27MW06, 15MW01, 15MW03, 27EW-01, 27EW-02, 27EW-04, 27EW-08, 27EW-12, 27EW-13, 27EW-14, 27EW-16, 27EW-17 and 27EW-18	Fall 2010
		Add TDS analysis to wells 27MW02 and 15MW02	Fall 2011
		Change sampling frequency from quarterly to semiannually	Winter 2012
	IR Site 28	Change sampling frequency for dissolved metals at wells 28SW03 and 28SW04 From quarterly to semiannually	Fall 2008
		Add quarterly sampling at well 28SW05	Spring 2008
		Change sampling frequency for dissolved metals at wells 28SW01 and 28SW02 from quarterly to annually	Spring 2009
		Discontinue sampling at IR Site 28	Summer 2009
		Discontinue groundwater elevation measurement at IR Site 28	Fall 2009
		Add sampling for arsenic and copper at wells 28SW02, 28SW03 and 28SW08	Fall 2012
		Discontinue analysis for VOCs	Fall 2013
NA	IR Site 35	Add sampling at wells AOC23-MW-01, AOC23-MW-02, AOC23-MW-03 and AOC23-MW-04	Fall 2009
		Discontinue groundwater elevation measurement at well MBG-04	Fall 2009
		Discontinue sampling at wells130AOC23-MW-01, 130AOC23-MW-02, 130AOC23-MW-03, and 130AOC23-MW-04	Fall 2012
		Discontinue groundwater elevation measurement	Fall 2013

Table 3-1.
BGMP Changes Summary (continued)

OU No.	IR Site No.	Changes	Implementation Schedule
NA	Sitewide	Perform basewide groundwater elevation measurement semiannually	Fall 2009
		Remove basewide groundwater elevation measurements for groundwater monitoring wells within sites removed from BGMP	Fall 2013

Notes:

BGMP = Basewide Groundwater Monitoring Program
DO = dissolved oxygen
IR Site = Installation Restoration Site
NAPs = natural attenuation parameters
NFA = no further action
ORP = oxygen reduction potential
OU = Operable Unit
PCB = polychlorinated biphenyl
pH = potential of hydrogen
RAWP = remedial action work plan
ROD = Record of Decision
SVOC = semi-volatile organic compounds
TDS = total dissolved solids
TPH = total petroleum hydrocarbon
VOCs = volatile organic compounds
WQ Parameters = water quality parameters including; pH, ORP, DO, temperature, and specific conductance

3.4 BASEWIDE GROUNDWATER MONITORING PROGRAM OPTIMIZATION

Each quarter, the BGMP will be analyzed for modification and optimization. The BGMP will continue to evaluate the effectiveness of the implemented remedies, and support site decisions, including no further action (NFA) and site closure. The components of the optimization process are briefly described below and are presented in more detail in Worksheet #17 of the SAP (Appendix A).

3.4.1 Target Analyte List Evaluation

The amount of data available from the IR/OU sites allows for trends to be evaluated using professional judgment to identify specific target analytes for the optimization process. Secondary decision criteria (cross-, up-, or down-gradient sentry wells, evaluation of ongoing remedial actions pursuant to the ROD, etc.) would be considered in addition to trend analyses for individual analytes or suites of analytes at each IR/OU site. As IR sites progress through the CERCLA process, the target analyte list for groundwater at each site will be evaluated as part of this BGMP.

3.4.2 Sampling Frequency

The frequency of wells sampled as part of the BGMP will be evaluated annually. The sampling frequency of each well should be sufficient to establish concentration trends and to evaluate any temporal variations. Trend analysis will be conducted by preparing and interpreting concentration versus time plots of the data. Mann-Kendall and Sen's method are broadly accepted statistical tools to analyze time-series groundwater monitoring data to quantitatively determine if the measured concentrations of a chemical are increasing, decreasing, or stable over time; and could provide supporting line of evidence to recommend or maintain ongoing MNA. In practice, trend lines are fitted to the most recent data points (typically six points) for each target analyte in a monitoring well. A well may have more than one sampling frequency for all its target analytes. This methodology is discussed in greater detail in Worksheet #17 of the SAP (Appendix A).

3.4.3 Well Decommissioning

Well decommissioning at Alameda Point is necessary to remove wells when they are no longer needed or required. This includes wells which monitor a plume that has a reducing trend with a defined footprint where the plume configuration has not changed; COCs have not been detected above RGs or above comparison criteria for at a minimum of two years previously; damaged or unusable wells; and wells that are no longer needed at the site based on remedial actions which have reduced contaminant concentrations (the success of which is evaluated under separate reports and task orders). Well decommissioning also reduces Navy environmental liabilities at Alameda Point, wells may be decommissioned at the discretion of the Navy. Wells that are candidates for decommissioning will be identified on the basis of the optimization analysis, site history, other investigations conducted by the Navy, future site use, and well physical conditions (i.e., damaged, etc.). The Navy may decommission wells throughout the duration of the BGMP as the opportunity arises. Wells that are decommissioned will be reported in the annual report.

3.5 PROBLEM STATEMENT

Previous investigations under CERCLA have documented releases of hazardous chemicals to subsurface groundwater at OUs and IR sites across the former base. Previous treatability studies and removal and remedial actions have addressed these sources of chemical release in soil and groundwater, and have significantly reduced the concentrations. The BGMP is designed to continue to monitor concentrations of the chemicals of concern at OUs and IR sites where remediation efforts have not been completed. The BGMP thus provides supplemental information for ongoing remedial action and monitored natural attenuation evaluations, and additional groundwater data for post-ROD. The problem definition is discussed in detail in Worksheet #10 of the SAP and the data quality objectives for the BGMP are discussed in detail in Worksheet #11 of the SAP.

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4 FIELD IMPLEMENTATION

This section summarizes the general site activities to be completed in support of the BGMP. These activities include permitting and notification, mobilization, utility clearance, groundwater sampling, soil gas sampling, hydropunch sampling, well installation, well maintenance and/or repair, well decommissioning, and surveying.

4.1 PERMITTING AND NOTIFICATION

Permits for the decommissioning of the identified wells will be acquired through the Alameda County Environmental Health Department. All well decommissioning activities will be performed under the guidance and supervision of a California Professional Geologist (PG), Certified Engineering Geologist (CEG), or Professional Engineer (PE) in accordance with California Department of Water Resources Bulletin 74-90, and any applicable local regulations.

Before the beginning of field work, the MMEC Group will inform the Navy of the nature and schedule of the planned field work. The MMEC Group will coordinate site access through the BRAC Caretaker Support Officer (CSO) or Activity Point of Contact (POC), and will notify the Resident Officer in Charge of Construction (ROICC) of the upcoming schedule for field work.

Coordination of sampling activities will take place between the MMEC Group and the Navy RPM regarding contractors performing work at individual IR Sites, prior to initiating the field sampling activities. Daily discussions between the field team leader, the quality officer, and the field team staff will take place prior to any sampling activities. These meetings will entail a discussion of assignments for the field team including which wells and what analytical groups will be sampled for the day. Additionally, end of day meetings will take place to catalogue that all assignments were implemented correctly.

Underground Service Alert (USA) will be notified of the intended activities at least 3 business days prior to the initiation of any invasive work. Alameda Utilities will also be notified prior to intrusion activities.

4.2 GROUNDWATER SAMPLING

To remain consistent with the current BGMP, groundwater samples will be collected from the groundwater monitoring wells located at various IR and OU sites at Alameda Point, as identified in the SAP (Appendix A). The methods by which groundwater sampling will be performed are outlined in the SAP (Appendix A). Wells that will be sampled are not identified specifically in this Work Plan and SAP. The BGMP is a fluid program and specific wells that will be sampled during each BGMP sampling event will be identified prior to that specific event and documented as an update to Worksheet #17 of the SAP. Analytical methods that may be used for groundwater samples throughout the course of the BGMP are identified in the SAP. The tasks in Sections 4.2.1 and 4.2.2 are associated with groundwater sampling.

4.2.1 Water Level Measurements

Measurements of groundwater levels will be collected only at the monitoring wells identified for sampling during each quarterly event. The water level will be measured immediately prior to sampling of each well during every sampling event.

4.2.2 Monitoring Well Sampling

Groundwater from monitoring wells will be sampled for offsite analysis by a Department of Defense and California-approved laboratory, as described in the SAP (Appendix A). Monitoring wells will be purged and sampled in accordance with U.S. EPA groundwater sampling operating procedure (U.S. EPA, 2010). The low-flow (*low-stress*) groundwater sampling purge rate is between 100 to 500 milliliters per minute (mL/min) with a preference for the lower end of this range during the initial stages of purging. The overall goal of low-flow purging is to minimize water table drawdown, and appropriate procedures will be followed during low-flow purging operations. A submersible pump (bladder pump) with a flow controller and disposable tubing and bladders will be used for sample collection. Passive groundwater sampling methods using a Snap Sampler may also be implemented. Sampling details are provided in the SAP (Appendix A).

4.3 SOIL GAS SAMPLING

Soil gas will be collected from wells identified in the SAP. The wells will be purged of 1 to 7 system volumes of “dead space” and leak checked prior to collection of the soil gas samples. Samples will be collected using Summa canisters at a flow rate not to exceed 200 mL/min. Soil gas samples will be analyzed using U.S. EPA Methods TO-3 and TO-15.

4.4 MOBILIZATION AND SITE PREPARATION

Prior to any intrusive activities, the MMEC Group Site Supervisor will identify the proposed wells to be decommissioned on the ground. Marking paint will be used to mark each proposed location. The MMEC Group will work with the Navy ROICC and Caretaker Site Office (CSO) to establish and identify appropriate laydown areas for all equipment, materials, and vehicles required to implement the field tasks.

4.5 UTILITY CLEARANCE

Prior to intrusive activities, each proposed drilling location for well installation will be cleared for utilities by a private utility-locating service, using handheld equipment, to ensure that no subsurface utilities are present. The MMEC Group will also notify USA and Alameda Utilities at least 72 hours prior to initiation of any subsurface drilling activities, including well decommissioning. All locations will be marked and cleared for subsurface drilling. A minimum distance of 2 feet from the closest observed utility will be maintained at all drilling locations; additionally, a minimum distance of 20 feet will be maintained from all overhead power lines.

4.6 WELL DECOMMISSIONING

Monitoring wells identified by the Navy for destruction will be decommissioned in accordance with California Department of Water Resources Bulletin 74-90, and any applicable local regulations. Well materials, including, but not limited to, protective surface casings, bollards, traffic boxes, polyvinyl chloride (PVC) or other riser pipe and/or screen, filter sand, bentonite seal material, annular seal, concrete, and/or cement fill material, will be removed and disposed of in accordance with the MMEC Group current waste management plan. The decommissioning of the wells will be performed under the guidance of a California-registered PG, CEG, or PE, by a drilling contractor with a valid State of California C-57 License.

4.7 WELL INSTALLATION

Well installation may be needed at Alameda Point as part of this BGMP in order to expand an existing well network or evaluate IR site conditions based on data collected under separate task orders and documented in other reports and planning documents. Installation of Navy-identified monitoring wells will be in accordance with the California Department of Water Resources Bulletin 74-90 and any applicable local regulations. The wells will be permitted through the local oversight agency, and access to the well installation locations will be coordinated through the Navy. Prior to any ground disturbance, the site will be marked with white paint (or other appropriate marking technique) and USA and Alameda Utilities will be notified at least 72 hours in advance of the planned disturbance. Additionally, the MMEC Group may decide to employ a private utility-locating service to clear the proposed disturbance area of underground utilities. Well materials, including, but not limited to, PVC or other riser pipe and/or screen, filter sand, bentonite seal material, annular seal, concrete, and/or cement fill material, will be installed in accordance with the permitting agency's guidelines, as well as in accordance with local, state, and federal regulations. The installation of the wells will be performed under the guidance of a California-registered PG, CEG, or PE, by a drilling contractor with a valid State of California C-57 License

4.8 HYDROPUNCH SAMPLING

Hydropunch sampling may be needed in order to evaluate groundwater conditions at IR sites in areas where a groundwater monitoring well may not exist. The locations of hydropunch samples will be determined based on data collected under separate task orders and documented in other planning documents or thru evaluation of groundwater data collected as part of the BGMP at the direction of the Navy. Hydropunch sampling will provide a means to evaluate groundwater conditions at IR sites without having to install a permanent monitoring well. Grab groundwater samples will be collected from borings advanced by DPT using a HydroPunch sampling tool or equivalent. Grab groundwater samples may be collected from any water bearing zone at Alameda if needed. Once the targeted sampling depth is reached, the HydroPunch tool will be threaded onto a steel drive pipe and pushed several feet into the saturated formation. The drive pipe will be pulled upward to open the inlet of the sampler, allowing groundwater to flow into the screened sample chamber. Samples will be collected from the sample chamber using a stainless steel or disposable bottom-emptying bailer, or peristaltic pump. Water quality readings (dissolved oxygen, turbidity, salinity, pH, etc.) will be taken before the sample is collected. Grab groundwater samples will be collected in the appropriate sample container based on the analytical method requirements.

4.9 WELL MAINTENANCE ACTIVITIES

Well maintenance activities, including repair or replacement of bolts, locks, J-plugs, well boxes (or components thereof), broken casings, and well monuments/pads, may be performed as part of the BGMP. The maintenance activities will be performed on wells that have been identified as requiring maintenance or repair through the BGMP. Repairs such as bolt, lock, or J-plug replacement may take place at the time of the observed deficiency. Repairs such as replacement of well boxes, well monument/pads, or broken casings will require proper notification and possible mobilization of contractors to perform the maintenance/repair work. All work will be performed in accordance with the California Department of Water Resources Bulletin 74-90 and with applicable local regulations, and will be properly permitted.

4.10 SURVEYING

Newly installed wells and wells that have been repaired, such that their elevations may have been altered, will be surveyed. Surveying activities will be conducted by a California-licensed land surveyor. The northings, eastings, and elevations will be collected with a minimum accuracy of ± 0.01 foot. Elevations will be surveyed relative to mean sea level (msl).

4.11 DECONTAMINATION

All sampling equipment will be thoroughly decontaminated prior to initiating any site work. Equipment will be (1) washed using potable water and a non-phosphate detergent, (2) rinsed with potable water, (3) rinsed with distilled or deionized water, and (4) allowed to air dry until its next use. Equipment decontamination areas will be located within or adjacent to the work areas. Decontamination liquids will be managed along with other IDW.

4.12 INVESTIGATION DERIVED WASTE

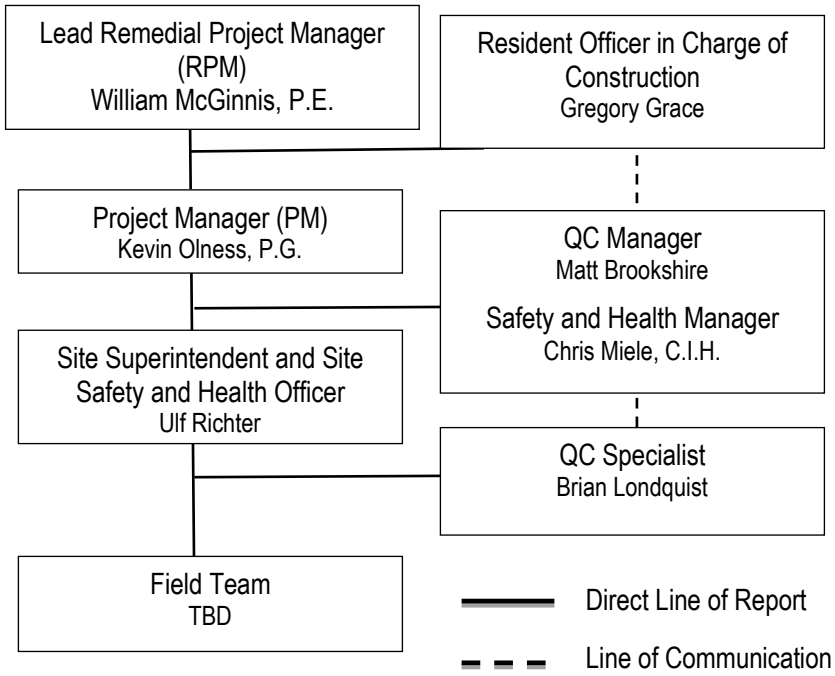
All IDW will be properly disposed of in accordance with U.S. EPA and California Department of Transportation (DOT) guidelines in the WMP (Appendix B). Waste may include wastewater from decontamination procedures, purged groundwater, soil cuttings, and used personal protective equipment (PPE). These wastes will be stored in 55-gallon steel drums and appropriately labeled until waste characterization is complete. Disposal of IDW will be performed within 90 days of generation. The IDW will be removed to an offsite disposal facility, and movement will be coordinated with the NAVFAC SW Remedial Project Manager (RPM) and the Alameda Point ROICC.

5 CONTRACTOR QUALITY CONTROL PLAN

The Contractor Quality Control (CQC) Plan outlined in this section has been developed to address construction-related quality control activities specific to this project. Additionally, QC activities related to project–specific sampling and analyses are addressed in the SAP (Appendix A).

5.1 CONTRACTOR QUALITY CONTROL ORGANIZATION

The CQC organizational structure and key members of the CQC organization for this project are presented in the following chart. The Field Quality Control (QC) Representation and Implementation Team will be identified before the start of fieldwork.



5.2 DUTIES, RESPONSIBILITIES, AND AUTHORITIES OF QC PERSONNEL

The various duties, responsibilities, and authorities of the key MMEC Group QC personnel are summarized in the organization chart and Sections 5.2.1 and 5.2.2.

Note that the subcontractor's CQC representative will also be identified and integrated into the overall QC activities. The responsibility and authority of the subcontractor's CQC representative will be the same as those of the MMEC Group Field QC Specialist.

5.2.1 Quality Control Manager

Matt Brookshire (Certified Manager of Quality/Operation Excellence, Certified Hazardous Materials Manager, Certified Professional in Storm Water Quality) is the QC Manager for this contract. Mr. Brookshire has 19 years of experience in various environmental engineering projects for federal agencies, local governments, and private clients. Mr. Brookshire has extensive experience in QC management of projects for field investigations, removal actions,

laboratory services, environmental compliance, and plan and report preparation (including work plans, sampling and analysis plans, field sampling plans, quality assurance project plans, site assessment reports, monitoring reports, and remedial investigation reports) for the Navy.

5.2.2 Site Superintendent

Ulf Richter has served multiple roles on complex and challenging environmental projects. Roles fulfilled by Mr. Richter include Construction Superintendent and Quality Manager, Field Manager, Site Safety and Health Officer, and Field Team Leader.

For 16 years, Mr. Richter has supported a variety of environmental investigations, remediation, environmental sampling, and compliance projects involving the oversight of multiple subcontractors and staff with zero recordable health and safety and environmental incidents.

Types of work conducted and overseen by Mr. Richter include, but are not limited to, direct push technology drilling, hollow-stem auger drilling, mud drilling, soil excavations, and collection of soil, groundwater, storm water, soil gas, sediment, air, and surface water samples. Mr. Richter has completed the Unites States Army Corps of Engineers (USACE) Construction Quality Management for Contractors training in April 2012, the Occupational Safety and Health Administration (OSHA) 30-hour and 10-hour training course in construction safety and health, the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) general site worker training, and HAZWOPER Supervisor/Site Safety Officer training.

5.3 QC DOCUMENTATION PROCEDURES

The section describes the daily CQC Report, documentation of site activities, and CQC submittals.

5.3.1 Daily Contractor Quality Control Report

The daily CQC Report will serve as the basic document for recording the daily CQC activities and will document the overall effectiveness of the QC program. The CQC Report Form (see Exhibit 1) provides detailed information regarding job site location, work performance, weather conditions, types and results of inspections performed, locations and descriptions of deficiencies, deficiencies corrected, and other comments. The CQC Report Form will be completed and signed by the QC Specialist. In general, The CQC Report Form provides the following QC information:

- Date and relevant contractual information of the project work.
- QC activities performed as part of the three-phase CQC control process (further discussion of the three-phase CQC process is presented in Section 5.5) for each Definable Feature of Work (DFOW).
- QC Checklist Forms (Exhibits 2 and 3) completed as part of the three-phase CQC control process for each DFOW.
- Rework items and the status of corrective actions implemented.
- Other related QC activities, such as meeting and/or inspections completed.
- Certification and test data reports in support of the QC activities.

5.3.2 Documentation of Site Activities

The following additional documents will be used for reporting onsite activities:

- **Site Safety Documentation:** Site safety documentation procedures are presented in the project Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP).
- **Daily Notes:** Internal daily notes will be kept for the site activities. These notes generally will be recorded on standard MMEC Group daily notes forms or in a bound notebook. Separate notebooks can be kept for different tasks. The notes will be recorded daily by the Site Superintendent or designee and will be used to prepare the daily CQC Report.
- **Contractor Production Report (CPR):** The Site Superintendent will submit a daily CPR (Exhibit 4) to the Navy or ROICC summarizing the work performed by the MMEC Group and its subcontractors, including a description of trades working on the project, number of personnel working, use of major equipment, and delivery of major items.
- **Meeting Minutes:** CQC meeting discussions will be documented in meeting minutes. Copies of these minutes will be attached to the CQC Report in accordance with their three-phase activity classification.

5.3.3 Contractor Control Submittals

The daily CQC Report and CPR are the primary CQC submittals required for this project.

5.4 LIST OF DEFINABLE FEATURES OF WORK

A DFOW is a work element that has discrete performance goals, can be separated from other tasks, and has distinct QC requirements. Based on the fieldwork scope, the following DFOWs are identified:

- Premobilization coordination meeting
- Mobilization
- Field work activities (groundwater sampling, soil gas sampling, etc.)
- Waste management and disposal
- Demobilization of field work

DFOWs will be managed in accordance with the specific performance requirements stated in this Work Plan through the implementation of a three-phase inspection approach, as discussed below.

5.5 PROCEDURES FOR PERFORMING THREE PHASES OF QUALITY CONTROL

The MMEC Group CQC Program will be based on the three phases of control stated in the Unified Facility Guide Specification for both onsite and offsite QC. In general, the three phases of control are performed in stages as each DFOW is implemented, as follows:

- **Preparatory Phase:** Involves QC activities performed prior to field implementation of the DFOW.
- **Initial Phase:** Includes the QC activities performed during the first day of DFOW field implementation.
- **Follow-Up Phase:** Involves QC activities performed daily for continuing DFOW until completed satisfactorily.

These three phases of control and inspection formats provide an effective methodology through the entire project to see that product quality is met at each stage of the DFOW. As a result, corrective actions, if required, can be identified and implemented quickly.

QC Checklists (Exhibits 2 and 3) for each phase are required for critical inspections and are completed during the inspection to document results. Checklists will be prepared for QC activities associated with each DFOW. These checklists are maintained by the site QC specialist and will become part of the inspection reports as specified in Section 5.5.1. QC inspection and test results are documented by the Site CQC Representative in daily CQC Reports. The QC inspection for each of the three phases of control is discussed in Sections 5.5.1 through 5.5.3.

5.5.1 Preparatory Inspections

Preparatory QC inspections are performed prior to beginning each DFOW. These inspections include review of submittal requirements and other contract requirements with supervisory personnel; verification of contract drawings and specifications so that provisions are made to provide required field control testing plans; examination of the work area to ascertain that the preliminary work has been completed; verification of field dimensions, lines, and grades; physical examination of materials and equipment; and review of the safety plan and appropriate activity hazard analysis. Other inspections include the following:

Receiving Inspection: Receiving inspections include a visual examination and accounting of materials obtained from suppliers upon arrival at the site. These inspections are performed to verify that the materials received meet design specifications, are free of defects, and have not been damaged in transit.

Calibration: Calibration of laboratory and field testing equipment is performed onsite and offsite using standards traceable to the National Bureau of Standards or other national standards, as appropriate. Calibration of equipment defines the accuracy of measurements taken and ensures that the instrumentation is in proper working condition.

Equipment Inspection: Equipment inspections are performed to verify that the equipment is in proper working order.

The inspection results will be documented using the Preparatory Phase Checklist (Exhibit 2) and reported in the daily CQC Report (Exhibit 1).

5.5.2 Initial QC Inspections

Initial QC inspections are performed when the DFOW begins in the field. The initial QC inspections include an examination of the initial quality of workmanship and a review of control testing for compliance with contract and Work Plan requirements. They see that safety standards are met and document conflicts between field implementation and the Work Plan. It is critical that conflicts are resolved quickly so that work can be continued without future issues. An initial QC inspection will be performed for each new crew working on the DFOW until the crew can achieve the acceptable quality standard required by the contract. The initial QC inspection is completed when such level of confidence is documented in daily CQC Reports. The initial QC inspection results will be documented using the Initial Phase Checklist (see Exhibit 3) and reported in the daily CQC Report (Exhibit 1).

5.5.3 Follow-Up QC Inspections

Follow-up QC inspections are performed daily or at appropriate intervals as the DFOW progresses to verify continuing compliance with contract requirements and safety standards. The follow-up QC inspections continue until completion of the DFOW. Follow-up QC inspections are performed to verify compliance with contract requirements, maintain the workmanship, and see that the rework items and QC testing are performed according to plan. Follow-up QC inspection reports are maintained by the site QC specialist. The follow-up QC inspection results will be provided in the daily CQC Report (Exhibit 1).

Documentation

All inspections will be a matter of record and will be documented on the inspection forms and maintained in accordance with CQC Plan requirements for documentation procedures. As discussed above, the CQC Report, including QC Checklists, will serve as the basic document for recording the daily CQC activities and will document the overall effectiveness of the QC Program. The daily CQC Report (Exhibit 1), CPR (Exhibit 4), and QC Checklist forms (Exhibits 2 and 3) will be filled out and signed by the site QC specialist.

Procedures to Complete Rework Items

During CQC activities, specific tasks not meeting Work Plan requirements will be identified as “rework items” that must be corrected before those tasks can be resumed. The QC Manager will maintain a list of rework items. The list will include information regarding the nonconformance or item that needs to be reworked, the date that the deficiency was discovered, and the date that the deficiency was corrected. If the correction was made on the same day that it was discovered, there is no requirement to report a rework item.

Nonconforming items and activities are those items or activities that do not meet project requirements. When such a condition is identified, the MMEC Group will implement a corrective action program to achieve the following:

- Document the nonconforming item or activity and determine the cause of nonconformance and its effect on project performance and the integrity of completed work.
- Correct or replace the nonconforming item in the most efficient and effective manner.
- Verify and document that the corrective action taken was successful.

Documentation of Nonconforming Items

The site QC specialist will document nonconformance items on the daily CQC Report to clearly state what is not in compliance, the date on which the noncompliance was originally discovered, and the date on which the work was corrected.

5.5.4 Implementation of Corrective Action

The MMEC Group will stop work on items or features pending satisfactory correction of deficiencies noted by MMEC Group project personnel or subcontractors. The Site Superintendent will have the authority to stop work until corrective actions are implemented. In some cases where the corrective action is obvious, it can be implemented immediately upon identification of the nonconformance. Other circumstances could require additional input from technical or operations staff, additional equipment and materials, or changes in existing structures or completed work. The site QC specialist will not allow work to be added to or built upon nonconforming work unless the Navy CQC Representative concurs that the correction can be made without stopping ongoing work. If the corrective action requires a revision or variance to the contract specification or drawing, such revision or variance must be approved by the Contracting Officer before it is implemented in the field.

5.5.5 Verification and Documentation of Corrective Action

Successful completion of corrective actions for nonconformance will be verified by the site QC specialist during a follow-up inspection. The daily CQC Report will reflect the corrective actions completed. Recurring nonconformance of a similar nature will be investigated to determine the root cause of the problem to eliminate or minimize future occurrences of the nonconformance. The status of rework items and corrective action will be documented using the Rework Items List form presented in Exhibit 5.

6 PROJECT SCHEDULE AND REPORTING

This section discusses the project schedule and reporting requirements of the BGMP.

6.1 PROJECT SCHEDULE

The MMEC Group will prepare and track progress on the project schedule and revise the schedule if major changes occur. Any problems experienced or anticipated will be communicated promptly to the Navy for assistance or resolution. A project schedule is presented on Figure 6.

6.2 REPORTING

The reporting for the BGMP will follow the same methods and procedures and provide the same information as in previous years. Reports will be prepared on an annual basis. The most recent annual report was prepared for the 2014 BGMP (SES TECH, 2015).

The annual reports will include, but not be limited to, the following:

- Introductory section providing a very brief discussion of the monitoring tasks performed that year. Tabulated tidal stages.
- Tabulated methane soil gas results.
- Tabulated analytical results.
- Tabulated groundwater levels for only the wells being sampled; other data collected will be placed in the appendices. Tabulated groundwater levels will be reviewed for any anomalous water elevations over the last 8 quarters.
- Graphical contaminant analytical results, indicating concentration as a function of time (trends).
- Program optimization summary.
- Recommendations.

As mentioned previously in this Work Plan, potentiometric maps will no longer be developed for each IR site. A groundwater flow directional arrow will be included on figures where information for individual IR sites is shown.

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7 REFERENCES

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FIGURES

Figure 1 Facility Location Map
Figure 2 Operable Unit and Installation Restoration Site Locations
Figure 3 Groundwater Monitoring Well Locations
Figure 4 Groundwater Monitoring Wells Sampled in 2014
Figure 5 Soil Gas Monitoring Locations
Figure 6 Project Schedule

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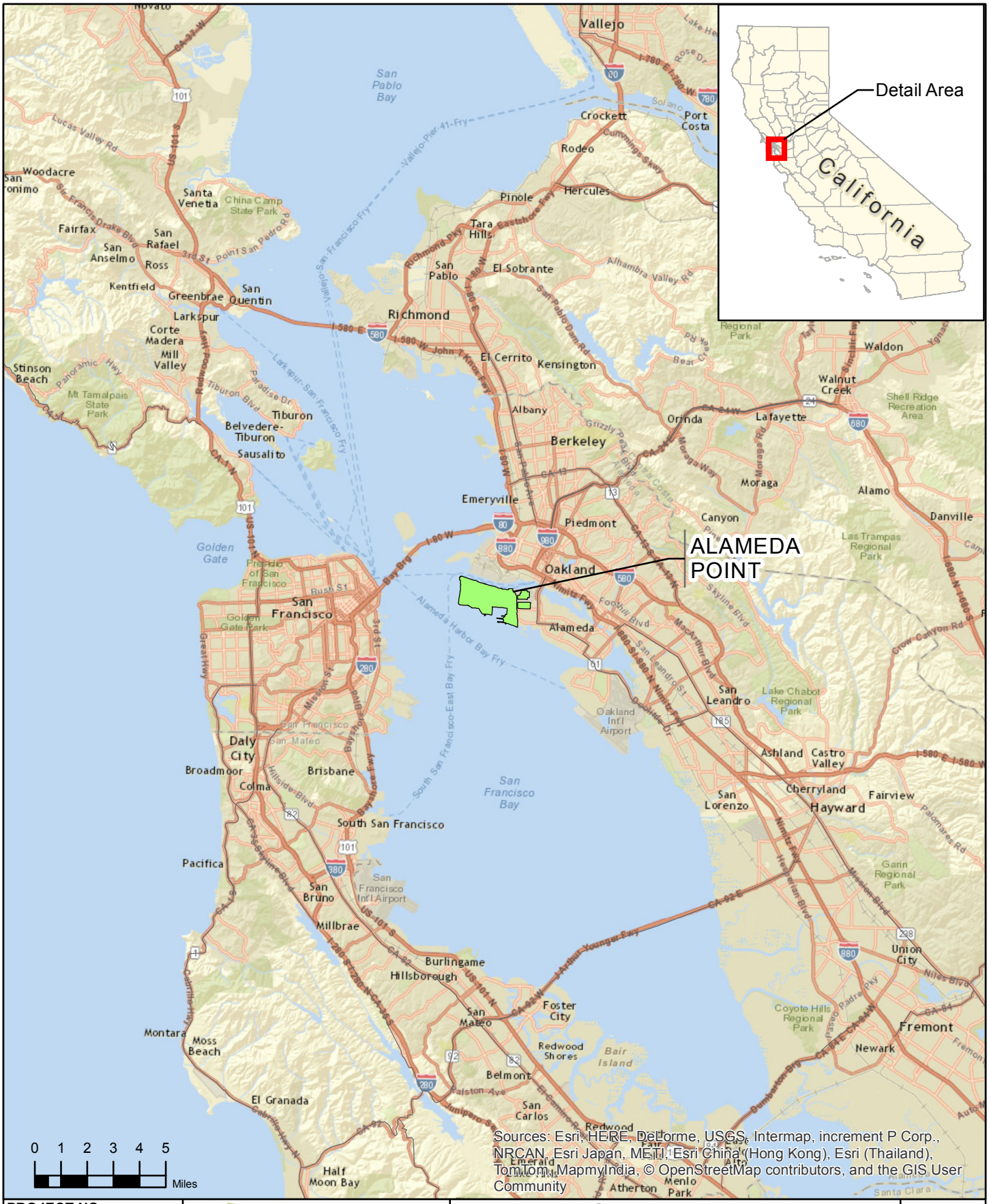

PROJECT NO.: 5023-14-6096	MNEC GROUP	Basewide Groundwater Monitoring Program Alameda Point, Alameda, California	FIGURE
DATE: FEBRUARY 2015		FACILITY LOCATION MAP	1
DRAWN BY: SB			
CHECKED BY: KO			

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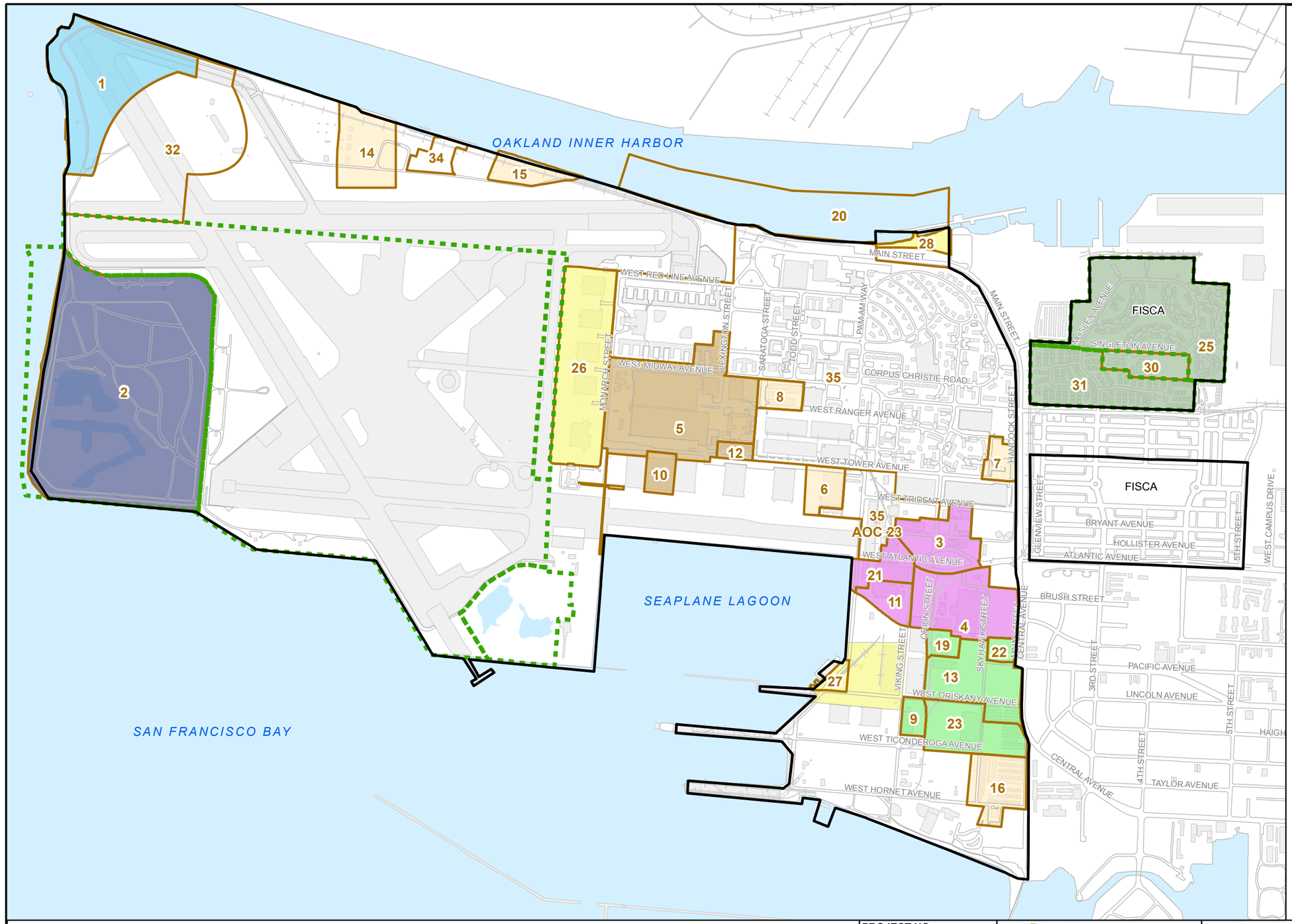

Legend

- Alameda Point Boundary
- Facility Infrastructure
- Railroad
- 1 Installation Restoration Site and Number
- Operable Unit 1
- Operable Unit 2A
- Operable Unit 2B
- Operable Unit 2C
- Operable Unit 3
- Operable Unit 4A
- Operable Unit 5
- Operable Unit 6
- Federal Transfer Parcel

Notes:

FISCA = Fleet and Industrial Supply Center Alameda

0 1,200 2,400 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OPERABLE UNIT AND INSTALLATION RESTORATION SITE LOCATIONS

FIGURE 2

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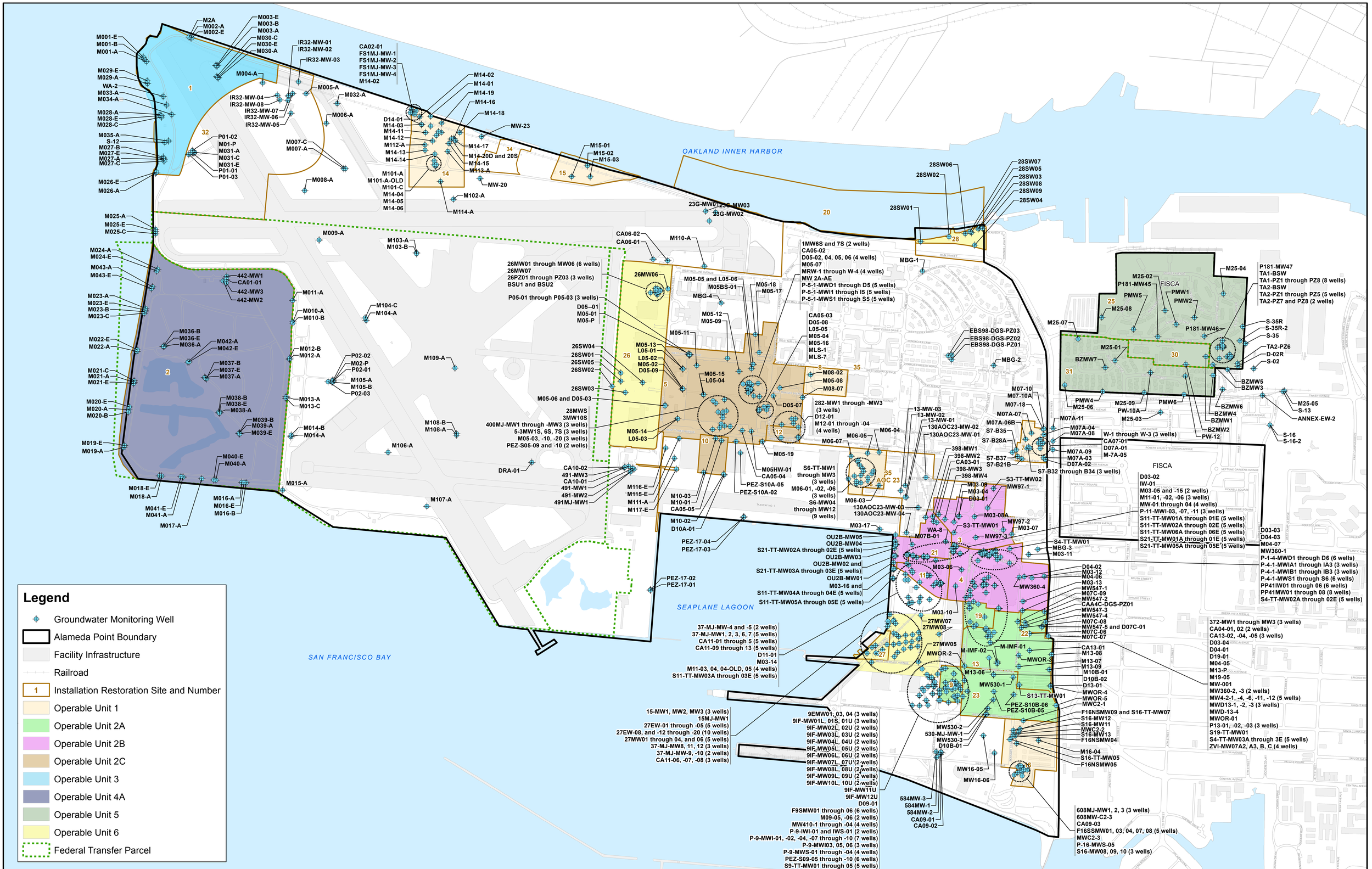


Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH (6 wells were not plotted on the map because coordinates were unavailable).
Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014.
Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.
FISC = Fleet and Industrial Supply Center Alameda

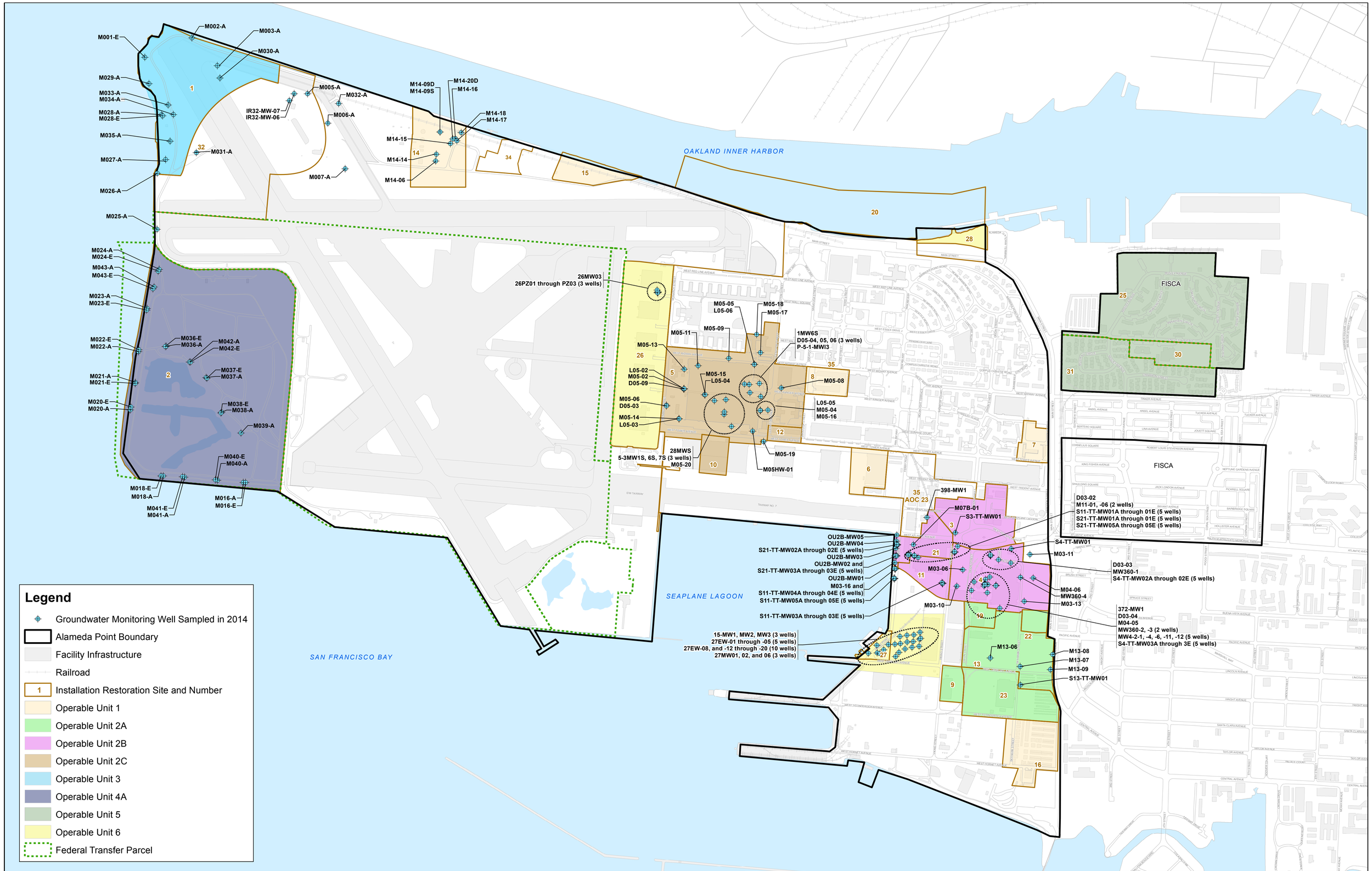
PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

MNEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

GROUNDWATER MONITORING WELL LOCATIONS

FIGURE 3



Notes:
Only the wells sampled during the 2014 Basewide Groundwater Sampling Program are shown. Refer to Figure A-3 for the locations of all wells.
Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014.
FISC = Fleet and Industrial Supply Center Alameda

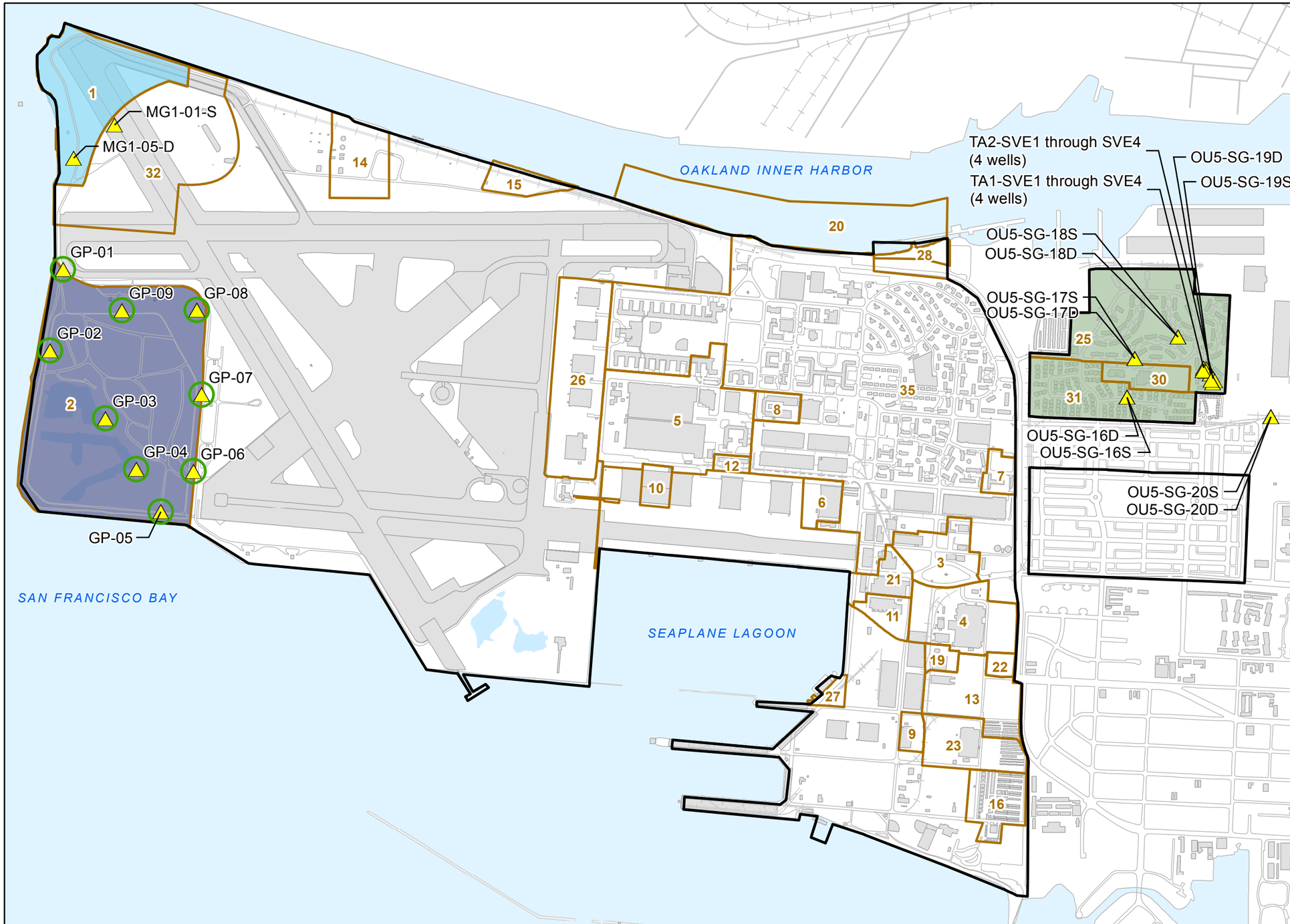
PROJECT NO.:	0023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

GROUNDWATER MONITORING WELLS SAMPLED IN 2014

FIGURE 4



Legend

- Soil Gas Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- Installation Restoration Site and Number
- Operable Unit 3
- Operable Unit 4A
- Operable Unit 5
- Facility Infrastructure

Notes:
All known and possibly existing soil gas monitoring wells are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes the report for monitoring conducted in 2014 (SES-TECH, 2015), and database information from 2007 for wells not monitored in 2014.
Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 1,200 2,400 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

SOIL GAS MONITORING LOCATIONS

FIGURE 5

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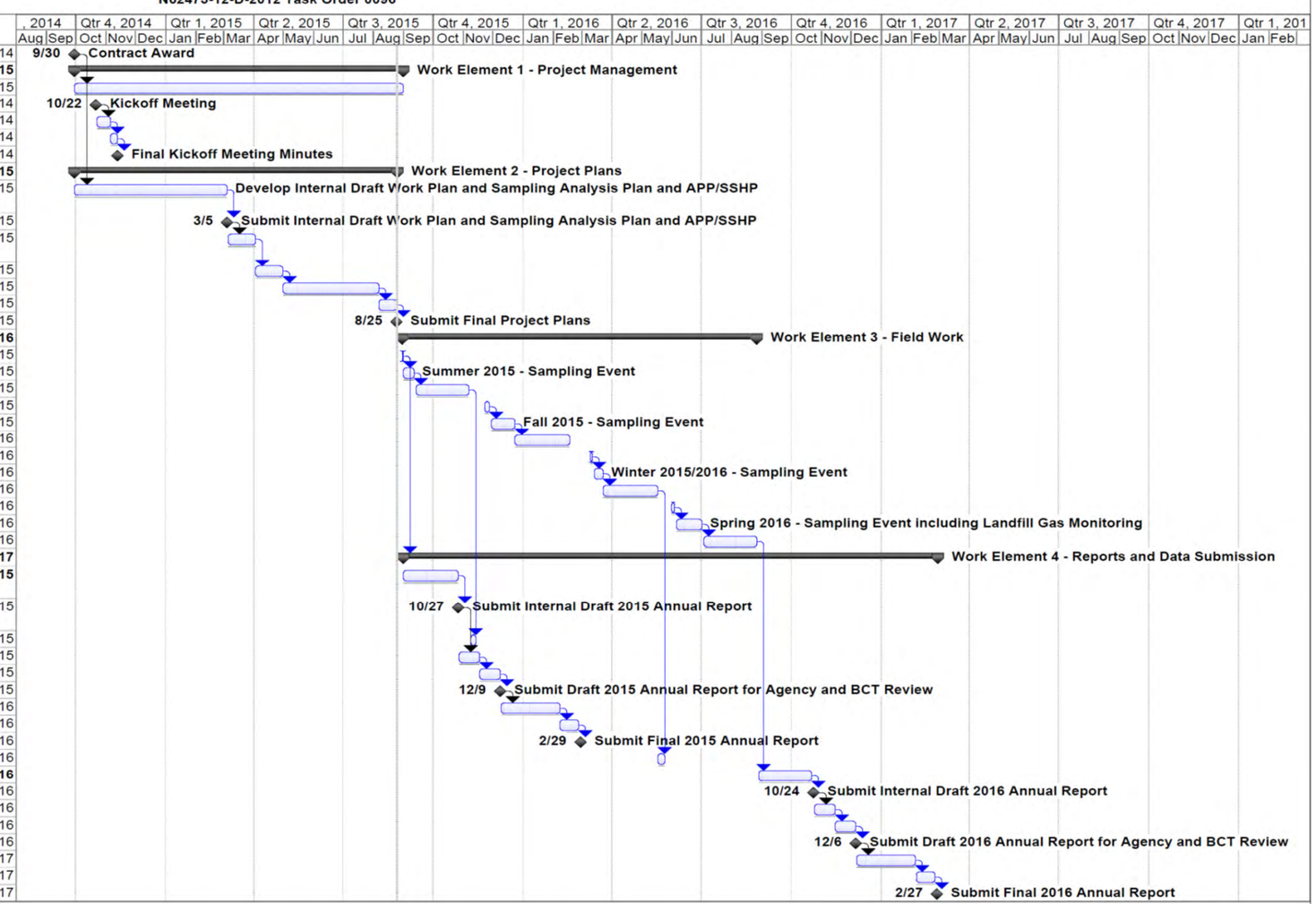
Proposed Project Schedule
Installation Restoration Sites
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California
N62473-12-D-2012 Task Order 0096

ID	Task Name	Duration	Start	Finish
1	Contract Award	0 days	Tue 9/30/14	Tue 9/30/14
2	**Work Element 1 - Project Management**	**240 days**	**Tue 9/30/14**	**Mon 8/31/15**
3	Project Management and Support	12 mons	Tue 9/30/14	Mon 8/31/15
4	Kickoff Meeting	1 day	Wed 10/22/14	Wed 10/22/14
5	Draft Kickoff Meeting Minutes	10 days	Thu 10/23/14	Wed 11/5/14
6	Navy Review - Draft Kickoff Meeting Minutes	5 days	Thu 11/6/14	Wed 11/12/14
7	Final Kickoff Meeting Minutes	3 days	Thu 11/13/14	Mon 11/17/14
8	**Work Element 2 - Project Plans**	**236 days**	**Tue 9/30/14**	**Tue 8/25/15**
9	Develop Internal Draft Work Plan and Sampling Analysis Plan and APP/SSHP	112 days	Tue 9/30/14	Wed 3/4/15
10	Submit Internal Draft Work Plan and Sampling Analysis Plan	1 day	Thu 3/5/15	Thu 3/5/15
11	Navy Review of Internal Draft Project Plans	20 days	Fri 3/6/15	Thu 4/2/15
12	Develop Draft Project Plans	20 days	Fri 4/3/15	Thu 4/30/15
13	Submit Draft Project Plans for Navy Review	70 days	Fri 5/1/15	Thu 8/6/15
14	Develop Final Project Plans	12 days	Fri 8/7/15	Mon 8/24/15
15	Submit Final Project Plans	1 day	Tue 8/25/15	Tue 8/25/15
16	**Work Element 3 - Field Work**	**260 days**	**Mon 8/31/15**	**Fri 8/26/16**
17	Summer 2015 - Mobilization	1 day	Mon 8/31/15	Mon 8/31/15
18	Summer 2015 - Sampling Event	9 days	Tue 9/1/15	Fri 9/11/15
19	Summer 2015 - Data Management	40 days	Mon 9/14/15	Fri 11/6/15
20	Fall 2015 - Mobilization	5 days	Mon 11/23/15	Fri 11/27/15
21	Fall 2015 - Sampling Event	18 days	Mon 11/30/15	Wed 12/23/15
22	Fall 2015 - Data Management	40 days	Thu 12/24/15	Wed 2/17/16
23	Winter 2015/2016 - Mobilization	2 days	Thu 3/10/16	Fri 3/11/16
24	Winter 2015/2016 - Sampling Event	7 days	Mon 3/14/16	Tue 3/22/16
25	Winter 2015/2016 - Data Management	40 days	Wed 3/23/16	Tue 5/17/16
26	Spring 2016 - Mobilization	3 days	Wed 6/1/16	Fri 6/3/16
27	Spring 2016 - Sampling Event including Landfill Gas Monitori	20 days	Mon 6/6/16	Fri 7/1/16
28	Spring 2016 - Data Management	40 days	Mon 7/4/16	Fri 8/26/16
29	**Work Element 4 - Reports and Data Submission**	**390 days**	**Tue 9/1/15**	**Mon 2/27/17**
30	**Develop Internal Draft 2015 Annual Report (begins after receipt of validated data from Spring 2015 event)**	**40 days**	**Tue 9/1/15**	**Mon 10/26/15**
31	Submit Internal Draft 2015 Annual Report	1 day	Tue 10/27/15	Tue 10/27/15
32	NIRIS Submittal - Spring and Summer 2015 Data	5 days	Mon 11/9/15	Fri 11/13/15
33	Navy Review of Internal Draft 2015 Annual Report	15 days	Wed 10/28/15	Tue 11/17/15
34	Develop Draft 2015 Annual Report	15 days	Wed 11/18/15	Tue 12/8/15
35	Submit Draft 2015 Annual Report for Agency and BCT Revie	1 day	Wed 12/9/15	Wed 12/9/15
36	Regulatory Agency and BCT Review - Draft 2015 Annual Re	60 edays	Wed 12/9/15	Sun 2/7/16
37	Develop Final 2015 Annual Report	15 days	Mon 2/8/16	Fri 2/26/16
38	Submit Final 2015 Annual Report	1 day	Mon 2/29/16	Mon 2/29/16
39	NIRIS Submittal - Fall 2015 and Winter 2015/2016 Data	5 days	Wed 5/18/16	Tue 5/24/16
40	**Develop Internal Draft 2016 Annual Report**	**40 days**	**Mon 8/29/16**	**Fri 10/21/16**
41	Submit Internal Draft 2016 Annual Report	1 day	Mon 10/24/16	Mon 10/24/16
42	Navy Review of Internal Draft 2016 Annual Report	15 days	Tue 10/25/16	Mon 11/14/16
43	Develop Draft 2016 Annual Report	15 days	Tue 11/15/16	Mon 12/5/16
44	Submit Draft 2016 Annual Report for Agency and BCT Revie	1 day	Tue 12/6/16	Tue 12/6/16
45	Regulatory Agency and BCT Review - Draft 2016 Annual Re	60 edays	Tue 12/6/16	Sat 2/4/17
46	Develop Final 2016 Annual Report	15 days	Mon 2/6/17	Fri 2/24/17
47	Submit Final 2016 Annual Report	1 day	Mon 2/27/17	Mon 2/27/17



Project:
Date: Tue 8/25/15

Task | Split | Progress | Milestone | Summary | Project Summary | External Tasks | External Milestone | Deadline

Page 1

PROJECT NO.: 5023-14-6096	MNEC GROUP	Basewide Groundwater Monitoring Program Alameda Point, Alameda, California	FIGURE
DATE: August 2015			
DRAWN BY: SWB		Project Schedule	6
CHECKED BY: KO			

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EXHIBITS

Exhibit 1 Contractor Quality Control Report
Exhibit 2 Preparatory Phase Checklist
Exhibit 3 Initial Phase Checklist
Exhibit 4 Contractor Production Report
Exhibit 5 Rework Items List

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EXHIBIT 1

CONTRACTOR QUALITY CONTROL REPORT

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CONTRACTOR QUALITY CONTROL REPORT

(ATTACH ADDITIONAL SHEETS IF NECESSARY)

DATE

REPORT NO

PHASE	CONTRACT NO	CONTRACT TITLE

PREPARATORY

WAS PREPARATORY PHASE WORK PREFORMED TODAY? YES ☐ NO ☐

IF YES, FILL OUT AND ATTACH SUPPLEMENTAL PREPARATORY PHASE CHECKLIST.

Schedule Activity No.	Definable Feature of Work	Index #

INITIAL

WAS INITIAL PHASE WORK PREFORMED TODAY? YES ☐ NO ☐

IF YES, FILL OUT AND ATTACH SUPPLEMENTAL INITIAL PHASE CHECKLIST.

Schedule Activity No.	Definable Feature of Work	Index #

FOLLOW-UP

WORK COMPLIES WITH CONTRACT AS APPROVED DURING INITIAL PHASE? YES ☐ NO ☐

WORK COMPLIES WITH SAFETY REQUIREMENTS? YES ☐ NO ☐

Schedule Activity No.	Description of Work, Testing Performed & By Whom, Definable Feature of Work, Specification Section, Location and List of Personnel Present

REWORK ITEMS IDENTIFIED TODAY (NOT CORRECTED BY CLOSE OF BUSINESS)

Schedule Activity No.	Description

REWORK ITEMS CORRECTED TODAY (FROM REWORK ITEMS LIST)

Schedule Activity No.	Description

REMARKS (Also Explain Any Follow-Up Phase Checklist Item From Above That Was Answered "NO"), Manuf. Rep On-Site, etc.

Schedule Activity No.	Description

AUTHORIZED QC MANAGER AT SITE DATE

GOVERNMENT QUALITY ASSURANCE REPORT

DATE

QUALITY ASSURANCE REPRESENTATIVE'S REMARKS AND/OR EXCEPTIONS TO THE REPORT

Schedule Activity No.	Description

GOVERNMENT QUALITY ASSURANCE MANAGER DATE

CONTRACTOR QUALITY CONTROL REPORT

(CONTINUATION SHEET)
(ATTACH ADDITIONAL SHEETS IF NECESSARY)

DATE	
REPORT NO.	

PHASE	CONTRACT NO	CONTRACT TITLE

FOLLOW-UP

WORK COMPLIES WITH CONTRACT AS APPROVED DURING INITIAL PHASE? YES ☐ NO ☐

WORK COMPLIES WITH SAFETY REQUIREMENTS? YES ☐ NO ☐

Schedule Activity No.	Description of Work, Testing Performed & By Whom, Definable Feature of Work, Specification Section, Location and List of Personnel Present

REMARKS (Also Explain Any Checklist Item From Above That Was Answered "NO"), Manuf. Rep. On-Site, etc.

Schedule Activity No.	Description

4296/2 (9/98)

EXHIBIT 2

PREPARATORY PHASE CHECKLIST

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PREPARATORY PHASE CHECKLIST

(CONTINUED ON SECOND PAGE)

CONTRACT NO	DEFINABLE FEATURE OF WORK	SPEC SECTION	DATE
		SCHEDULE ACT NO.	INDEX #

PERSONNEL PRESENT

GOVERNMENT REP NOTIFIED HOURS IN ADVANCE: YES ☐ NO ☐

NAME	POSITION	COMPANY/GOVERNMENT

SUBMITTALS

REVIEW SUBMITTALS AND/OR SUBMITTAL REGISTER. HAVE ALL SUBMITTALS BEEN APPROVED? YES ☐ NO ☐

IF NO, WHAT ITEMS HAVE NOT BEEN SUBMITTED?

ARE ALL MATERIALS ON HAND? YES ☐ NO ☐

IF NO, WHAT ITEMS ARE MISSING?

CHECK APPROVED SUBMITTALS AGAINST DELIVERED MATERIAL. (THIS SHOULD BE DONE AS MATERIAL ARRIVES.)

COMMENTS:

MATERIAL STORAGE

ARE MATERIALS STORED PROPERLY? YES ☐ NO ☐

IF NO, WHAT ACTION IS TAKEN?

SPECIFICATIONS

REVIEW EACH PARAGRAPH OF SPECIFICATIONS.

DISCUSS PROCEDURE FOR ACCOMPLISHING THE WORK.

CLARIFY ANY DIFFERENCES.

PRELIMINARY WORK & PERMITS

ENSURE PRELIMINARY WORK IS CORRECT AND PERMITS ARE ON FILE.

IF NOT, WHAT ACTION IS TAKEN?

PREPARATORY PHASE CHECKLIST

(CONTINUED FROM FIRST PAGE)

CONTRACT NO	DEFINABLE FEATURE OF WORK	SPEC SECTION	DATE
		SCHEDULE ACT NO.	INDEX #

TESTING

IDENTIFY TEST TO BE PERFORMED, FREQUENCY, AND BY WHOM.

WHEN REQUIRED?

WHERE REQUIRED?

REVIEW TESTING PLAN.

HAS TEST FACILITIES BEEN APPROVED?

SAFETY

ACTIVITY HAZARD ANALYSIS APPROVED? YES ☐ NO ☐

REVIEW APPLICABLE PORTION OF EM 385-1-1.

MEETING COMMENTS

NAVY/ROICC COMMENTS DURING MEETING.

OTHER ITEMS OR REMARKS

OTHER ITEMS OR REMARKS:

QC MANAGER DATE

EXHIBIT 3

INITIAL PHASE CHECKLIST

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INITIAL PHASE CHECKLIST

		SPEC SECTION	DATE
CONTRACT NO	DEFINABLE FEATURE OF WORK	SCHEDULE ACT NO.	INDEX #

PERSONNEL PRESENT

GOVERNMENT REP NOTIFIED _____ HOURS IN ADVANCE: YES ☐ NO ☐

NAME	POSITION	COMPANY/GOVERNMENT

PROCEDURE COMPLIANCE

IDENTIFIY FULL COMPLIANCE WITH PROCEDURES IDENTIFIED AT PREPARATORY. COORDINATE PLANS, SPECIFICATIONS, AND SUBMITTALS.

COMMENTS:

PRELIMINARY WORK

ENSURE PRELIMINARY WORK IS COMPLETE AND CORRECT. IF NOT, WHAT ACTION IS TAKEN?

WORKMANSHIP

ESTABLISH LEVEL OF WORKMANSHIP.

WHERE IS WORK LOCATED?

IS SAMPLE PANEL REQUIRED? YES ☐ NO ☐

WILL THE INIITAL WORK BE CONSIDERED AS A SAMPLE? YES ☐ NO ☐

(IF YES, MAINTAIN IN PRESENT CONDITION AS LONG AS POSSIBLE AND DESCRIBE LOCATION OF SAMPLE)

RESOLUTION

RESOLVE ANY DIFFERENCES.

COMMENTS:

CHECK SAFETY

REVIEW JOB CONDITIONS USING EM 385-1-1 AND JOB HAZARD ANALYSIS

COMMENTS:

OTHER

OTHER ITEMS OR REMARKS

QC MANAGER DATE

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EXHIBIT 4

CONTRACTOR PRODUCTION REPORT

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CONTRACTOR PRODUCTION REPORT

(ATTACH ADDITIONAL SHEETS IF NECESSARY)

		DATE
CONTRACT NO	TITLE AND LOCATION	REPORT NO
CONTRACTOR	SUPERINTENDENT	
AM WEATHER	PM WEATHER	MAX TEMP (F) / MIN TEMP (F)

WORK PERFORMED TODAY

Schedule Activity No.	WORK LOCATION AND DESCRIPTION	EMPLOYER	NUMBER	TRADE	HRS

JOB SAFETY

WAS A JOB SAFETY MEETING HELD THIS DATE? (If YES attach copy of the meeting minutes)	☐ YES	☐ NO	TOTAL WORK HOURS ON JOB SITE, THIS DATE, INCL CON'T SHEETS
WERE THERE ANY LOST TIME ACCIDENTS THIS DATE? (If YES attach copy of completed OSHA report)	☐ YES	☐ NO	CUMULATIVE TOTAL OF WORK HOURS FROM PREVIOUS REPORT
WAS CRANE/MANLIFT/TRENCHING/SCAFFOLD/HV ELEC/HIGH WORK/ HAZMAT WORK DONE? (If YES attach statement or checklist showing inspection performed.)	☐ YES	☐ NO	TOTAL WORK HOURS FROM START OF CONSTRUCTION
WAS HAZARDOUS MATERIAL/WASTE RELEASED INTO THE ENVIRONMENT? (If YES attach description of incident and proposed action.)	☐ YES	☐ NO	

Schedule Activity No.	LIST SAFETY ACTIONS TAKEN TODAY/SAFETY INSPECTIONS CONDUCTED	☐ SAFETY REQUIREMENTS HAVE BEEN MET.

EQUIPMENT/MATERIAL RECEIVED TODAY TO BE INCORPORATED IN JOB (INDICATE SCHEDULE ACTIVITY NUMBER)

Schedule Activity No.	Submittal #	Description of Equipment/Material Received

CONSTRUCTION AND PLANT EQUIPMENT ON JOB SITE TODAY. INDICATE HOURS USED AND SCHEDULE ACTIVITY NUMBER.

Schedule Activity No.	Owner	Description of Construction Equipment Used Today (incl Make and Model)	Hours Used

Schedule Activity No.	REMARKS

CONTRACTOR/SUPERINTENDENT DATE

CONTRACTOR PRODUCTION REPORT (CONTINUATION SHEET)		DATE
CONTRACT NO	TITLE AND LOCATION	REPORT NO

WORK PERFORMED TODAY

Schedule Activity No.	WORK LOCATION AND DESCRIPTION	EMPLOYER	NUMBER	TRADE	HRS

Schedule Activity No.	LIST SAFETY ACTIONS TAKEN TODAY/SAFETY INSPECTIONS CONDUCTED

EQUIPMENT/MATERIAL RECEIVED TODAY TO BE INCORPORATED IN JOB (INDICATE SCHEDULE ACTIVITY NUMBER)

Schedule Activity No.	Submittal #	Description of Equipment/Material Received

CONSTRUCTION AND PLANT EQUIPMENT ON JOB SITE TODAY. INDICATE HOURS USED AND SCHEDULE ACTIVITY NUMBER.

Schedule Activity No.	Owner	Description of Construction Equipment Used Today (incl Make and Model)	Hours Used

Schedule Activity No.	REMARKS

INCLUDE ALL PERSONNEL WORK HOURS IN THE WORK PERFORMED SECTION ON THIS SHEET
INTO THE FRONT CONTRACTOR PRODUCTION REPORT

EXHIBIT 5

REWORK ITEMS LIST

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REWORK ITEMS LIST

Contract No. and Title:

Contractor:

NUMBER	DATE IDENTIFIED	DESCRIPTION	CONTRACT REQUIREMENT (Spec. Section and Par. No., Drawing No. and Detail No., etc.)	ACTION TAKEN BY QC MANAGER	RESOLUTION	DATE COMPLETED

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Appendix A
SAMPLING AND ANALYSIS PLAN

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FINAL

SAMPLING AND ANALYSIS PLAN

(Field Sampling Plan and Quality Assurance Project Plan)

BASEWIDE GROUNDWATER MONITORING PROGRAM

ALAMEDA POINT, ALAMEDA, CALIFORNIA

August 2015

Prepared for:

Department of the Navy

Naval Facilities Engineering Command Southwest
1220 Pacific Highway
San Diego, California 92132-5190

Prepared by:

Multimedia Environmental Compliance Group
9177 Sky Park Court
San Diego, California 92123

Prepared Under:

Contract Number: N62473-12-D-2012
Task Order Number: 0096
Document Control Number (DCN): MMEC-2012-0096-0003

SAP WORKSHEET #1 TITLE AND APPROVAL PAGE

FINAL

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)

August 2015

Basewide Groundwater Monitoring Program

Alameda Point, Alameda, California

Prepared for:

DEPARTMENT OF THE NAVY
Naval Facilities Engineering Command Southwest
1220 Pacific Highway
San Diego, California 92132-5190

Prepared by:

MMEC Group
9177 Sky Park Court
San Diego, California 92123

Prepared Under:

Contract Number: N62473-12-D-2012
Task Order Number: 0096
Document Control Number (DCN): MMEC-2012-0096-0003

Review Signature: _____ 8/19/15
Matt Brookshire — Date
MMEC Group Quality Control Manager

NAVFAC Chemist Review Signature: SOLOMON.JUDITH.A.1461885000 Digitally signed by SOLOMON.JUDITH.A.1461885000 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=SOLOMON.JUDITH.A.1461885000 Date: 2015.08.20 13:31:59 -04'00'
NAVFAC Chemist — Date

Approval Signature: _____ _____
Joseph Michalowski — Date
NAVFAC SW Quality Assurance Officer

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EXECUTIVE SUMMARY

The Multimedia Environmental Compliance Group (MMEC Group), a joint venture between Amec Foster Wheeler Environment & Infrastructure, Inc. (Amec Foster Wheeler) and KMEA, is supporting the Naval Facilities Engineering Command Southwest (NAVFAC SW) by managing and conducting the Basewide Groundwater Monitoring Program (BGMP) at former Naval Air Station Alameda (Alameda Point), Alameda, California. This work is being performed under NAVFAC SW Contract Number N62473-12-D-2012, Task Order Number 0096.

The primary objective of this BGMP is to provide ongoing support via continued monitoring of groundwater and soil gas from various Installation Restoration (IR) and Operable Unit (OU) sites at Alameda Point. This monitoring is to help provide continuing data to support remedial actions and trend analysis under the Record of Decision (ROD) for the site. This Sampling and Analysis Plan (SAP) includes elements for routine soil gas monitoring, groundwater monitoring, well installation, well repair, well decommissioning, hydropunch sampling, surveying, and reporting. The SAP also provides for optimization and efficiency of the BGMP by describing a dynamic work strategy to allow for changes in the monitoring network and sampling approach.

Groundwater samples may be collected and analyzed for:

- Total Petroleum Hydrocarbon (TPH-e) (as diesel, jet fuel, and motor oil) by United States Environmental Protection Agency (U.S. EPA) Method 8015B
- TPH-p (as gasoline) by U.S. EPA Method 8015B
- Volatile Organic Compounds (VOCs) by U.S. EPA Method 8260B
- 1,4-Dioxane by U.S. EPA Method 8270 Selective Ion Monitoring (SIM)
- Semi-Volatile Organic Compounds (SVOCs) by U.S. EPA Method 8270C
- Polynuclear Aromatic Hydrocarbons (PAHs) by U.S. EPA Method 8310
- Pesticides by U.S. EPA Method 8081
- Polychlorinated Biphenyls (PCBs) by U.S. EPA Method 8082
- Dissolved Metals by U.S. EPA Methods 6010B/6020A/7470A
- Hexavalent Chromium by U.S. EPA Method 7196A
- Cyanide by U.S. EPA Method 335.2
- Dissolved Gases (methane, ethane, ethene) by standard operating procedure (SOP) RSK 175
- Anions (nitrate, nitrite, chloride, sulfate) by U.S. EPA Method 300.0
- Alkalinity by U.S. EPA Method 310.2
- Sulfide by U.S. EPA Method 376.2
- Ferrous Iron by Hach Test Kit
- Total Dissolved Solids (TDS) by U.S. EPA Method 150.1
- Biological Oxygen Demand (BOD) by U.S. EPA Method 405.1
- Chemical Oxygen Demand (COD) by U.S. EPA Method 410.4
- Total Organic Carbon (TOC) by U.S. EPA Method 415.1
- Dissolved Organic Carbons (DOC) by Standard Methods for the Examination of Water and Wastewater (SM) 5310C
- Census Deoxyribonucleic Acid (DNA) by Quantitative Real-Time Polymerase Chain Reaction [qPCR]
- Gross Alpha, Gross Beta by U.S. EPA Method 9310/900.0
- Radium-226 by U.S. EPA Method 9315/903.0
- Tritium by U.S. EPA Method 906.0
- Total Radium by U.S. EPA Method 904.0

EXECUTIVE SUMMARY (CONTINUED)

- Strontium-90 by U.S. EPA Method 905.0
- Gamma Emitting Radionuclides (Cs-137 and Co-60) by U.S. EPA Method 901.1
- Uranium Isotopes by U.S. EPA Method 908 Modified
- Water Quality (WQ) parameters:
  - Oxidation Reduction Potential (ORP) by field meter
  - pH by field meter
  - Dissolved Oxygen (DO) by field meter and Hach Test Kit
  - Temperature by field meter
  - Specific Conductance by field meter

Soil gas will be sampled annually and analyzed for:

- Fixed gases by U.S. EPA Method TO-03
- VOCs (including methane, ethane, and ethane) by U.S. EPA Method TO-15

Optimization for the increase or reduction in sampling frequency, addition or removal of analytical suites, addition or removal of wells, or addition or removal of Installation Restoration (IR)/Operable Unit (OU) sites to the BGMP will follow a multi-faceted approach. This approach recognizes site-specific decisions and data needs and includes feedback from the various Remedial Project Managers (RPMs) who oversee the remedial design and implementation, and regulatory agencies. BGMP optimization changes will be summarized in the annual BGMP reports.

Additional activities, including hydropunch sampling, well installation, well maintenance and/or repair, well decommissioning, and surveying, may be performed at various IR sites. Hydropunch sampling, well installation, and decommissioning will be performed by a licensed driller with a valid C-57 water well license, and under the guidance of a licensed California Professional Geologist, Certified Engineering Geologist, or Professional Engineer. Well maintenance and/or repairs will be performed to ensure compliance with local, state, and federal regulations regarding well condition, and will be performed by qualified individuals. Surveying will be performed by a state-licensed land surveyor, and well components will be surveyed to known datum.

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Attachment 2: Laboratory Certifications
Attachment 3: Laboratory Standard Operating Procedures

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ABBREVIATIONS AND ACRONYMS

°C	degrees Celsius
µg/L	micrograms per liter
A	analysis
AHA	Activity Hazard Analysis
Amec Foster Wheeler	Amec Foster Wheeler Environment & Infrastructure, Inc.
amsl	above mean sea level
APP	Accident Prevention Plan
ASTM	American Society for Testing and Materials
BCT	BRAC Cleanup Team
BEC	BRAC Environmental Coordinator
BGMP	Basewide Groundwater Monitoring Program
bgs	below ground surface
BHC	benzene hexachloride
BOD	biological oxygen demand
BRAC	Base Realignment and Closure
BSU	Bay Sediment Unit
BTEX	benzene, toluene, ethylbenzene, xylene
CA	Corrective Action
CADD	computer-aided design (dynamic)
CAS	Chemical Abstracts Service
CCB	continuing calibration blank
CCC	criteria continuing concentration
CCV	continuing calibration verification
CEG	Certified Engineering Geologist
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CHMM	Certified Hazardous Materials Manager
CO	carbon monoxide
CO_2	carbon dioxide
Co-60	Cobalt-60
COC	chain of custody
COC	chemical of concern
COD	coefficient of determination
COD	chemical oxygen demand
COPC	chemicals of potential concern
CPR	cardiopulmonary pulmonary resuscitation
Cs-137	Cesium-137
CSM	conceptual site model

ABBREVIATIONS AND ACRONYMS (CONTINUED)

CSO	Caretaker Site Office
DCC	daily calibration check
DCN	document control number
DNA	Deoxyribonucleic Acid
DNAPL	dense non-aqueous phase liquid
DO	dissolved oxygen
DOC	dissolved organic compound
DoD	U.S. Department of Defense
DON	U.S. Department of the Navy
DOT	Department of Transportation
DQI	Data Quality Indicator
DQO	data quality objective
DTSC	Department of Toxic Substances Control
DTW	depth to water
EB	equipment blank
ELAP	Environmental Laboratory Accreditation Program
EWI	Environmental Work Instruction
ft	foot or feet
FWBZ	First Water Bearing Zone
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
GIS	geographic information system
H&S	health and safety
HAZWOPER	hazardous waste operations and emergency response
HSZ	hydrostratigraphic zone
IC	ion chromatography
ICAL	initial calibration
ICB	initial calibration blank
ICS	interference check solution
ICV	initial calibration verification
ID	identification
IDW	investigation-derived waste
IR	Installation Restoration
IS	internal standard
LCS	laboratory control sample
LDC	Laboratory Data Consultants, Inc.

ABBREVIATIONS AND ACRONYMS (CONTINUED)

LEL	lower explosive limit
L/min	liters per minute
LNAPL	light non-aqueous phase liquid
LOD	limit of detection
LOQ	limit of quantitation
LTM	Long-term Monitoring
LUC	Land Use Control
MCL	maximum contaminant level
MDC	minimum detected concentration
MDL	Method Detection Limit
MEC	munitions and explosive of concern
mg/L	milligrams per liter
MGP	manufactured gas plant
mL	milliliter
mL/min	milliliters per minute
mm	millimeters
MM/DD/YYYY	month/day/year
MMEC Group	Multimedia Environmental Compliance Group
MNA	Monitored Natural Attenuation
MS	mass spectrometer
MS	matrix spike
MSD	matrix spike duplicate
msl	mean sea level
MTBE	methyl tert-butyl ether
mV	millivolts
N_2	nitrogen
NA	not available/not applicable
NAPL	non-aqueous phase liquid
NAVFAC SW	Naval Facilities Engineering Command Southwest
NEDD	Navy Electronic Data Deliverable
NFA	No Further Action
NFESC	Naval Facilities Engineering Service Center
NIRIS	Naval Installation Restoration Information Solution
NPL	National Priorities List
NTU	Nephelometric turbidity unit
O_2	oxygen
OPS	Operating Properly and Successfully
ORP	oxidation reduction potential
OSHA	Occupational Safety and Health Administration

ABBREVIATIONS AND ACRONYMS (CONTINUED)

OU	Operable Unit
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Level
PARCCS	Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity
PCB	polychlorinated biphenyl
PCE	tetrachloroethene
pCi/L	picoCuries per Liter
P.E.	Professional Engineer
P.G.	Professional Geologist
pH	potential for hydrogen
PID	photoionization detector
PM	Project Manager
PMO	Program Management Office
POC	point of contact
PPE	personal protective equipment
QA	quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	quality control
QCM	Quality Control Manager
QL	Quantitation Limit
qPCR	Quantitative Real-Time Polymerase Chain Reaction
QSM	quality systems manual
RAD	radiological
RF	response factor
RG	Remedial Goal
RL	reporting limit
ROD	Record of Decision
ROICC	Resident Officer in Charge of Construction
RPD	relative percent difference
RPM	Remedial Project Manager
RPS	revolutions per second
RSD	relative standard deviation
S	sampling
S&A	sampling and analysis
SAP	Sampling and Analysis Plan
SB	source blank

ABBREVIATIONS AND ACRONYMS (CONTINUED)

SES-TECH	Sealaska Environmental Services, LLC – Tetra Tech EC, Inc.
SIM	Selective Ion Monitoring
SM	Standard Methods for the Examination of Water and Wastewater
SME	sporadic marginal exceedance
SOP	standard operating procedure
SPCC	system performance-check compound
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SVOC	semi-volatile organic compound
SWBZ	Second Water Bearing Zone
TB	trip blank
TBD	to be determined
TDS	total dissolved solids
TOC	total organic carbon
TPH	total petroleum hydrocarbon
TPH-e	total petroleum hydrocarbons extractable
TPH-g	total petroleum hydrocarbons as gasoline
TPH-p	total petroleum hydrocarbons purgeable
USA	Underground Service Alert
U.S. EPA	United States Environmental Protection Agency
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
VA	Veterans Affairs
VOA	volatile organic analyte
VOC	volatile organic compound
Water Board	California Regional Water Quality Control Board – San Francisco Region
WBZ	Shallow Water-Bearing Zone
WQ	water quality
WS	Worksheet

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SAP WORKSHEET #2 SAP IDENTIFYING INFORMATION

Site Name/Number:	Alameda Point, Alameda, California
Operable Unit:	Not Applicable
Contractor Name:	Multimedia Environmental Compliance Group (MMEC Group)
Contract Number:	N62473-12-D-2012
Contract Title:	Multimedia Environmental Compliance Engineering Support Services Contract
Work Assignment Number (optional):	Basewide Groundwater Monitoring Program Not Applicable
DCN:	MMEC-2012-0096-0001

- This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. EPA, 2005) and *EPA Guidance for Quality Assurance Project Plans,* EPA QA/G-5, QAMS (U.S. EPA, 2002).

This SAP is also prepared based on the requirements provided in the following documents:

- Department of Defense (DoD). *Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.
- Department of the Navy (DON). *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*, October 2009.
- NAVFAC Southwest (NAVFAC SW). Environmental Work Instruction (EWI) #1, Chemical Data Validation, November 2001; Environmental Work Instruction #6, Environmental Data Management and Required Electronic Delivery Standards, April 2005; Department of the Navy Environmental Restoration Program Manual, August 2006; Environmental Work Instruction #3, Selecting an Environmental Laboratory that Meets Environmental Restoration Program Requirements, August 2010; and Environmental Work Instruction #2, Review, Approval, Revision, and Amendment of Sampling Analysis Plans (SAPs), January 2011.
- Naval Facilities Engineering Service Center (NFESC). *Installation Restoration Chemical Data Quality Manual*, 1999.
- U.S. EPA. *Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Compliance with Other Laws Manual*, Draft Guidance, EPA/540/G-89/006. Office of Emergency and Remedial Response, Washington, DC, August 1988; *Guidance for the Data Quality Objectives (DQO) Process,* EPA QA/G-4, Final, September 1994; *Test Methods for Evaluation of Solid Wastes,* SW-846, Update III, 1997; *Guidance for the DQOs Process.* Office of Environmental Information, EPA/600/R-96/055. EPA QC/G-4. August 2000; *Guidance on Systematic Planning Using the DQO Process,* EPA QA/G-4. Office of Environmental Information EPA/240/B-06/001, February 2006. *National Functional Guidelines for Inorganic Superfund Data Review,* January 2010. *National Functional Guidelines for Organic Superfund Data Review,* October 2013.

SAP WORKSHEET #2 CONTINUED

1. Identify regulatory program: Basewide Groundwater Monitoring Program.
2. This SAP is an installation wide SAP.
3. List dates of scoping sessions that were held:

Scoping Session	Date
Alameda Basewide Groundwater Monitoring Technical Meeting	December 17, 2014

4. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation

Title	Date
Final Sampling and Analysis Plan (Field Sampling Plan/Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*02/2004*
Final Addendum 02 to the Sampling and Analysis Plan Field Sampling Plan/Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*10/2004*
Final Sampling and Analysis Plan – Addendum 3 (Field Sampling and Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*06/2009*
Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*08/2010*
Addendum 1 to the Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*12/2010*
Final Addendum 2 to the Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*11/2011*
Final Addendum 3 to the Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*11/2012*

SAP WORKSHEET #2 CONTINUED

Title	Date
Final Addendum 4 to the Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan), Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California	*01/2014*

5. List organizational partners (stakeholders) and connection with lead organization:

   U.S. EPA, California Department of Toxic Substances Control (DTSC), and California Regional Water Quality Control Board – San Francisco Region (Water Board) are regulatory stakeholders for this project.

   These regulatory agencies are not connected to the Navy or to NAVFAC SW.

6. Lead organization (see Worksheet [WS] #7 for detailed list of data users):

   NAVFAC SW

7. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

   - No required information omitted.

SAP WORKSHEET #2 CONTINUED

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	
2	Table of Contents SAP Identifying Information	
3	Distribution List	
4	Project Personnel Sign-Off Sheet	
Project Organization		
5	Project Organizational Chart	
6	Communication Pathways	
7	Personnel Responsibilities and Qualifications Table	
8	Special Personnel Training Requirements Table	
Project Planning/Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	
10	Problem Definition, Site History, and Background Site Maps (historical and present)	
11	Site-Specific Project Quality Objectives	
12	Measurement Performance Criteria Table	
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	
14	Summary of Project Tasks	
15	Reference Limits and Evaluation Table	
16	Project Schedule/Timeline Table	
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	
18	Sampling Locations and Methods/Standard Operating Procedure (SOP) Requirements Table Sample Location Map(s)	
19	Analytical Methods/SOP Requirements Table	
20	Field Quality Control Sample Summary Table	
21	Project Sampling SOP References Table Sampling SOPs	
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	
24	Analytical Instrument Calibration Table	
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	

SAP WORKSHEET #2 CONTINUED

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving, and Disposal Sample Handling Flow Diagram	
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain of Custody Form and Seal	
Quality Control Samples		
28	Quality Control (QC) Samples Table Screening/Confirmatory Analysis Decision Tree	
Data Management Tasks		
29	Project Documents and Records Table	
30	Analytical Services Table Analytical and Data Management SOPs	
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	
32	Assessment Findings and Corrective Action Responses Table	
33	Quality Assurance (QA) Management Reports Table	
D. Data Review		
34	Verification (Step I) Process Table	
35	Validation (Steps IIa and IIb) Process Table	
36	Validation (Steps IIa and IIb) Summary Table	
37	Usability Assessment	

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SAP WORKSHEET #3 DISTRIBUTION LIST

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address
Bill McGinnis, Professional Engineer (P.E.)	Lead RPM	NAVFAC SW BRAC PMO West	(619) 524-5811	william.mcginnis1@navy.mil 33000 Nixie Way, Bldg 50, 2nd Deck San Diego, CA 92147
Cecily Sabedra	Navy BRAC Environmental Coordinator (BEC), Alameda	NAVFAC SW BRAC PMO West	(619) 532-0972	cecily.sabedra@navy.mil 33000 Nixie Way, Bldg 50, 2nd Deck San Diego, CA 92147
Doug DeLong	BRAC Caretaker Site Office or Activity Point of Contact (POC)	NAVFAC SW BRAC PMO West	(510) 772-8832	douglas.delong@navy.mil 1 Ave of the Palms, Suite 161 San Francisco, CA 94130
Joseph Michalowski, Ph.D.	Quality Assurance Officer (QAO)	NAVFAC SW	(619) 532-4125	joseph.michalowski@navy.mil 1220 Pacific Highway Building 128, Mail Room San Diego, CA 92132 Attn: Code EVR JM
Gregory Grace	Resident Officer in Charge of Construction (ROICC)	Alameda Point	(510) 521-8709	gregory.grace@navy.mil Alameda Point 950 West Mall Square Suite-160 MS-2 Alameda, CA 94501
Diane Silva	Records Manager	NAVFAC SW	(619) 556-1280	diane.silva@navy.mil Command Records Manager ATTN: Code EV33 1220 Pacific Highway (NBSD Bldg. 3519) San Diego, CA 92132
Jim Hogan, P.E.	Program Manager	MMEC Group	(858) 514-6460	jim.hogan@amecfw.com 9177 Sky Park Court San Diego, CA 92123
Kevin Olness, Professional Geologist (P.G.)	Project Manager (PM)	MMEC Group	(619) 204-8195	Kevin.olness@amecfw.com 800 North Bell Ave Pittsburgh, PA 15106
Ulf Richter	Site Health and Safety Officer (SSHO) and Site Supervisor	MMEC Group	(619) 451-6200	urichter@kmea.net 9177 Sky park Court San Diego, CA 92123

SAP WORKSHEET #3 CONTINUED

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address
Lansana Coulibaly, Ph.D., P.E.	Senior Technical Lead	MMEC Group	(858) 633-2795	lansana.coulibaly@amecfw.com 9177 Sky park Court San Diego, CA 92123
Matthew Brookshire, CMQ/OE	Quality Control Manager (QCM)	MMEC Group	(858) 633-2793	matthew.brookshire@amecfw.com 9177 Sky park Court San Diego, CA 92123
William Jones	Project Manager	Blaine Tech Services, Inc.	(408) 639-0420	bjones@blainetech.com 1680 Rogers Ave San Jose, CA 95112
David Alltucker	Laboratory PM	Test America	(916) 374-4383	David.alltucker@testamericainc.com 880 Riverside Parkway West Sacramento, CA 95605
Ms. Linda Rauto	Operations Manager	Laboratory Data Consultants, Inc. (LDC)	(760) 634-0437	lrauto@lab-data.com 7750 El Camino Real, Ste. 2L Carlsbad, CA 92009

SAP WORKSHEET #4 PROJECT PERSONNEL SIGN-OFF SHEET

Name[1]	Organization/ Title/Role	Telephone Number (Optional)	Signature/E-mail Receipt[2]	SAP Section Reviewed	Date SAP Read
Kevin Olness, P.G.	MMEC Group PM	(619) 204-8195		All Worksheets	
Lansana Coulibaly, Ph.D., P.E.	MMEC Group, Senior Technical Lead	(858) 633-2795		All Worksheets	
Ulf Richter	SSHO, Site Supervisor	(619) 451-6200		All Worksheets	
William Jones	Blaine Tech Services, Inc.	(408) 639-0420		Worksheets #1, 3, 12, 15, 19, 20, 22, 23, 24, 25, 26, 27, 28, 30, 34, 35	
David Alltucker	Test America Project Manager	(619) 374-4383		Worksheets #15, 19, 23, 24, 25, 26, 28, 30, 34, 35, 36	
Linda Rauto	LDC Operations Manger	(760) 634-0437		Worksheets #1, 3, 12, 15, 19, 20, 22, 23, 24, 25, 28, 30, 34, 35, 36	

Notes:

1. Matthew Brookshire, MMEC Group QCM, and Joseph Michalowski, NAVFAC SW QAO, have reviewed and approved this SAP; therefore, their names are not listed above in the project personnel sign-off worksheet.
2. Copy of the complete sign-off sheet will be maintained in the project file.

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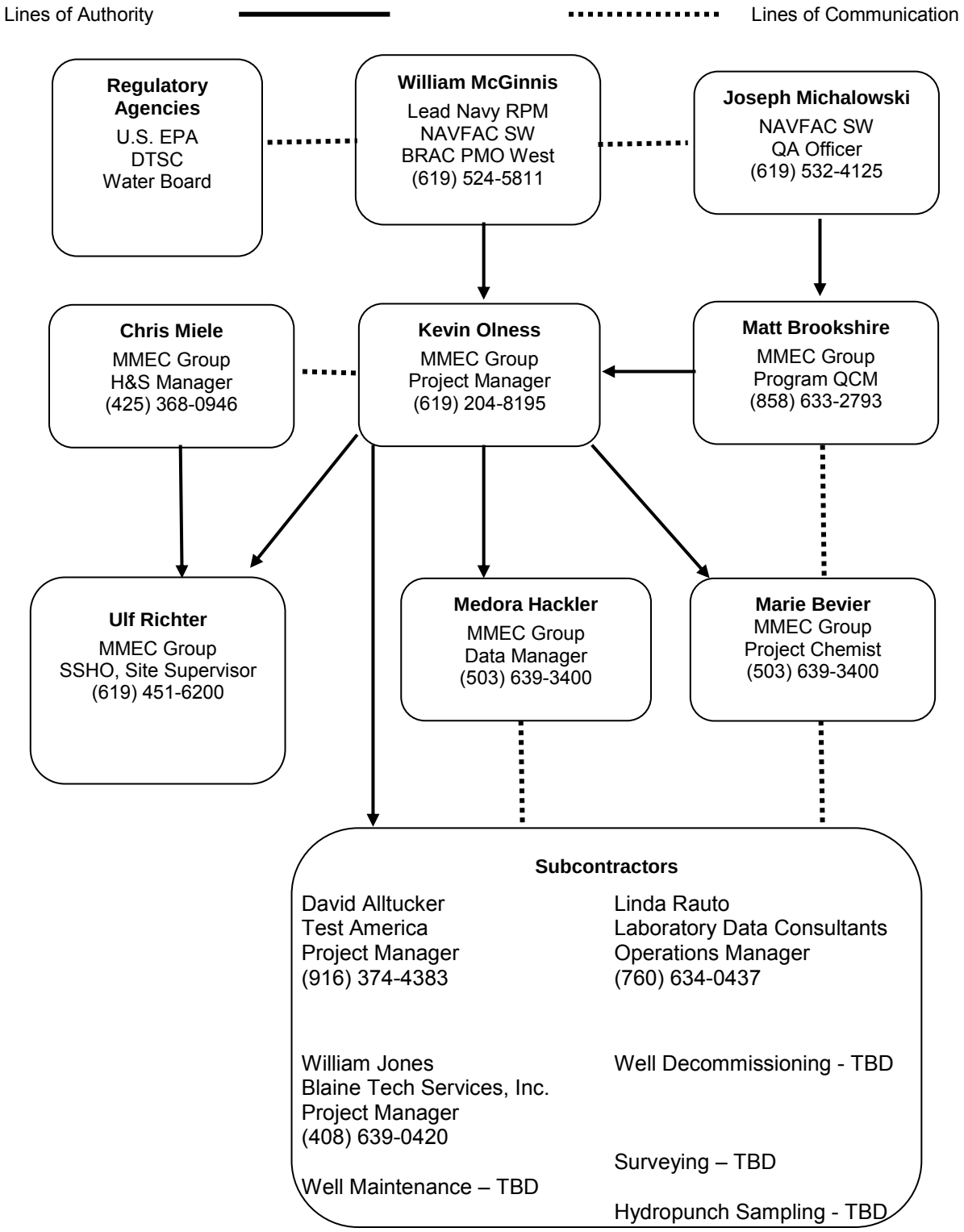
SAP WORKSHEET #5 PROJECT ORGANIZATIONAL CHART



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SAP WORKSHEET #6 COMMUNICATION PATHWAYS

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure (Timing, Pathway to and from, etc.)
SAP Amendment	MMEC Group	QCM: Matt Brookshire	(858) 633-2793 matthew.brookshire@amecfw.com	The MMEC Group QCM will submit all SAP amendments electronically to the NAVFAC SW QAO for review and approval prior to regulatory review and/or field implementation (see NAVFAC SW EWI #2). Major or significant changes will be documented in SAP amendments.
Field Change Request	MMEC Group	PM: Kevin Olness	(619) 204-8195 kevin.olness@amecfw.com	The MMEC Group PM will document any deviation from the SAP, including minor changes, by notifying the MMEC Group QCM by telephone and e-mail within 24 hours (see NAVFAC SW EWI #2). The NAVFAC SW RPM and QAO will be notified of field changes and of changes relating to site characterization that may entail a change in sampling location or analyses. All completed field changes will be documented in the final report.
Field Audit	MMEC Group	QCM: Matt Brookshire	(858) 633-2793 matthew.brookshire@amecfw.com	The MMEC Group QCM may conduct a field audit during project field work. The audit report will be maintained in project and QA files. Any issues requiring corrective action will be documented and assigned an appropriate response period. Problems with data quality will be reported to the MMEC Group PM by telephone and e-mail within 24 hours.

SAP WORKSHEET #6 CONTINUED

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure (Timing, Pathway to and from, etc.)
Stop Work	MMEC Group	PM: Kevin Olness SSHO: Ulf Richter	(619) 204-8195 kevin.olness@amecfw.com (619) 451-6200 urichter@kmea.net	The MMEC Group PM, QCM, or Site Safety and Health Officer, ROICC, CSO, or NAVFAC SW RPM and QAO may stop work in response to any serious quality- or safety-related issue, if warranted. The PM will communicate work stoppages to the project organization and NAVFAC SW PM by telephone and e-mail within 24 hours.
	NAVFAC SW	NAVFAC SW RPM: Bill McGinnis NAVFAC SW QAO: Joseph Michalowski	(619) 532-0902, (619) 532-0907 william.mcginnis1@navy.mil (619) 532-4125 joseph.michalowski@navy.mil	
Reporting of Health and Safety (H&S) Issues	MMEC Group	PM: Kevin Olness	(619) 204-8195 kevin.olness@amecfw.com	All H&S issues involving an injury, a “near miss,” or a condition that may result in an incident must be reported to the MMEC Group H&S Manager and the MMEC Group PM immediately. The PM will notify the NAVFAC SW RPM of serious H&S incidents/issues within 24 hours of occurrence. Non-serious incidents/issues may be forwarded to the NAVFAC SW RPM through the PM on a monthly basis via the monthly progress reports.

SAP WORKSHEET #6 CONTINUED

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure (Timing, Pathway to and from, etc.)
Notification of Non-Usable Analytical Data	MMEC Group	QCM: Matt Brookshire	(858) 633-2793 matthew.brookshire@amecfw.com	If significant problems are identified by the laboratory or the project team that impact the usability of the data (i.e., the data are rejected or the DQOs are not met), the MMEC Group QCM and the PM will notify the NAVFAC SW RPM by telephone and e-mail within 24 hours or the next business day.

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SAP WORKSHEET #7 PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS TABLE

Name	Title/Role	Organizational Affiliation	Responsibilities
William McGinnis	Lead RPM	NAVFAC SW	• Serves as lead project management representative for NAVFAC SW. • Verifies that work is accomplished as required by project scope of work. • Oversees project cost and schedule. • Provides formal technical instructions to MMEC Group staff. • Serves as the lead interface between agencies involved.
Joseph Michalowski	QAO	NAVFAC SW	• Reviews and approves SAP for compliance with NAVFAC SW and UFP-QAPP requirements. • Provides Navy oversight of MMEC Group's QA Program. • Provides technical and administrative oversight of MMEC Group's surveillance audit activities. • Serves as point of contact for QA and DON's Laboratory QA Program issues. • Coordinates training for improving the generation and maintenance of quality data. • Holds the authority to suspend project activities if QA requirements are not met.
Kevin Olness	PM	MMEC Group	• Coordinates work activities for the MMEC Group and subcontractor personnel and ensures that field activities are being performed in accordance with the Work Plan, SAP, Accident Prevention Plan (APP), and Site Safety and Health Plan (SSHP). • Promotes a safe work environment for all project personnel by applying work guidelines as specified in the SSHP and all applicable Occupational and Safety Health Administration (OSHA) regulations. • Oversees project, financial, schedule, and technical day-to-day management of the project. • Assists appropriate NAVFAC SW technical personnel in decision making when necessary.
Matt Brookshire	QCM	MMEC Group	• Ensures that project-specific SAP conforms to current NAVFAC SW and UFP-QAPP requirements. • Reviews and approves SAPs and work plans. • Communicates with PM and notifies PM of deviations from the SAP and nonconformance issues. • Coordinates field and laboratory QA and oversees field QA and project QA compliance. • Holds the authority to suspend project activities if QA requirements are not met.

SAP WORKSHEET #7 CONTINUED

Name	Title/Role	Organizational Affiliation	Responsibilities
Ulf Richter	SSHO, Site Supervisor	MMEC Group	• Ensures that all field activities are done per the SAP, APP, and SSHP. • Provides direction to field staff and subcontractors.
Marie Bevier	Project Chemist	MMEC Group	• Coordinates with the selected contracted laboratory. • Verifies appropriateness of sampling procedures, analytical methods, and laboratory quality systems. • Coordinates with the laboratory for field supplies, schedule, sample shipping, and deliverables. • Oversees data quality review and QA data validation deliverables.
David Alltucker	Laboratory QA Manager/ Project Manager	TestAmerica	• Is responsible for communication between the laboratory and MMEC Group. • Performs the analytical services per the project-specific SAP. • Ensures that the analysis is performed in accordance with specifications and meets the requirements of this SAP. • Reviews the laboratory data package before it is delivered to the MMEC Group.
Linda Rauto	Operations Manager	LDC	• Conducts independent, third-party validation of analytical data received from the laboratory per the project-specific SAP. • Assures the data end users of known and documented data quality.
William Jones	Project Manager	Blaine Tech Service, Inc.	• Coordinates with MMEC Group Project Manager for scheduling or field staff • Ensures sampling services provided meet all requirements of the SAP.

SAP WORKSHEET #8 SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

All field personnel will be required to have completed the Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste Emergency Response Operations (HAZWOPER) Standard Protection training as described in Title 29 Code of Federal Regulations (CFR) Section 1910.120, have completed the continued 8-hour HAZWOPER refresher, and submit to annual medical surveillance, as required by OSHA. Field personnel who directly supervise employees engaged in hazardous waste operations will also receive at least 8 hours of specialized supervisor training. The supervisor training covers Health and Safety (H&S) program requirements, training requirements, personal protective equipment (PPE) requirements, the spill containment program, and health-hazard monitoring procedures and techniques. At least one of the two field personnel will maintain cardiopulmonary pulmonary resuscitation (CPR) and standard first aid training certificates.

Safety requirements are addressed in detail in the SSHP, prepared separately. Copies of certifications and training records for team members and supervisors will be kept in the MMEC Group project file.

The subcontractor laboratory to be selected for this project will have successfully completed the laboratory evaluation process as described in the DoD Quality Systems Manual (QSM). Copies of their current laboratory certifications will be included in Attachment 2.

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SAP WORKSHEET #9 PROJECT SCOPING SESSION PARTICIPANTS

Project Name: Basewide Groundwater Monitoring Program	**Site Name: Installation Restoration Sites**
Projected Date(s) of Sampling: August 2015 – August 2016	**Site Location: Alameda Point, Alameda, California**
BRAC, NAVFAC SW RPM: Bob Sevcik	

Date of Session: Wednesday, October 22, 2014, at 10:00
Scoping Session Purpose: Kickoff Meeting

Name	Title/Role	Affiliation	Phone #	E-mail Address
Bill McGinnis	Lead RPM	NAVFAC SW	(619) 532-0907	william.mcginnis1@navy.mil
Bob Sevcik	RPM	NAVFAC SW	(619) 532-0902	robert.sevcik.ctr@navy.mil
Gregory Grace	ROICC	NAVFAC SW	(510) 521-8709	gregory.grace@navy.mil
Robert Perricone	ROICC	NAVFAC SW	(510) 521-8600	robert.perricone@navy.mil
Doug DeLong	Caretaker Site Office (CSO)	NAVFAC SW	(510) 772-8832	douglas.delong@navy.mil
Matt Brookshire	QC Manager	MMEC Group	(858) 633-2793	matthew.brookshire@amecfw.com
Kevin Olness	Project Manager	MMEC Group	(619) 204-8195	kevin.olness@amecfw.com
Curtis Moss	Technical Support	MMEC Group	(858) 869-7548	curtis.moss@amecfw.com
Don Kubik	Health & Safety Lead	MMEC Group	(510) 663-4115	don.kubic@amecfw.com
Lansana Coulibaly	Technical Lead	MMEC Group	(858) 633-2795	lansana.coulibaly@amecfw.com
Brian Londquist	Field Support	MMEC Group	(858) 300-4340	brian.londquist@amecfw.com

Notes:

Mr. Olness began by identifying the contract and task order number, and introduced the project team. Mr. Olness said that he is as the MMEC Group Project Manager, Jim Hogan is the MMEC Group Program Manager, Lansana Coulibaly is the Technical Lead, and Matt Brookshire is the QC Manager for the MMEC Group. Project roles and oversight agencies were identified and points of contact were discussed.

Health and safety was discussed, focusing on the need for the Navy and Marine Corps Public Health Center to review the APP/SSHP and associated documents. Additionally, Mr. Kubik indicated that he will be reviewing the APP/SSHP and all Activity Hazard Analyses (AHAs) to ensure compliance with the newly updated 2014 EM 385-1-1 manual. The APP/SSHP will be treated as a living document to accommodate changing field conditions. Hazards identified from previous groundwater monitoring events were identified and discussed.

Project management items, including acknowledgement of the conference call constituting the kickoff meeting for the delivery order, were discussed. It was agreed that an additional meeting (a pre-construction/field readiness meeting) will be held prior to the initiation of field activities in summer 2015. This meeting will include MMEC project staff, including the PM, MMEC Group subcontractors, and the Navy ROICC, CSO, and PM. Weekly communications will take place

SAP WORKSHEET #9 CONTINUED

between Mr. Olness and Mr. Sevcik, with no specific schedule. Monthly progress reports will be submitted with the invoices, and draft MPRs sent by the 15th of each month.

The Work Plan and the SAP will be a single document, with the SAP being an appendix to the Work Plan. The SAP will incorporate as much as possible regarding potential project tasks and optimization techniques, as this will serve as the primary sampling document for the next three years. That being said, the SAP needs to provide for some flexibility for sampling and/or analysis, and will be uploaded to Naval Installation Restoration Information Solution (NIRIS) for NAVFAC SW QAO review, which currently takes approximately 30 days. Quarterly, tables will be produced indicating any changes or proposed changes to the SAP (including addition/subtraction of wells, increased/decreased sampling frequency, sampling only constituents of concern for a particular IR site), and these tables will also need to be reviewed and approved by NAVFAC SW QAO.

The optimization will be an ongoing process, evaluated quarterly, based on the data that have been collected and are available for review. The proposed changes must meet the Long-Term Monitoring goals (concentrations below remedial goals (RGs) for at least 1 year, requirements of work plans, and other applicable decision documents), but are not necessarily subject to BRAC Cleanup Team (BCT) review and approval. The MMEC Group may want to re-evaluate some site conceptual models where treatment might not have achieved its goals.

Sealaska Environmental Services, LLC – Tetra Tech EC, Inc. (SES-TECH) is currently drafting the Annual Basewide Groundwater Monitoring Report, with an internal draft due in November 2014 (a copy of which will be provided to MMEC Group when available) and a draft version due in January 2015. The MMEC Group SAP should incorporate optimization recommendations from the SES-TECH report, as long as the recommendations meet the Record of Decision (ROD) and Long-term Monitoring (LTM) requirements. MMEC Group will review the last 5 years of groundwater data and perform optimization on an individual IR site basis, with optimization recommendations included in the annual report. Part of the reporting optimization is to no longer include the groundwater elevation contour maps, as is recommended in the SES-TECH report, which will be formally presented to the BCT in January 2015. A new report style that will be a concise data presentation with recommended changes and improvements/optimizations is suggested. The closeout strategies will be site specific in accordance with the ROD and other CERCLA documentation. These strategies will be produced by others as standalone documents, and are not part of this delivery order.

The field work will begin in summer 2015, likely in August, so that the sampling conforms with previous events, and is anticipated to last 7 work days. It is estimated to take approximately 40 work days to receive and validate the analytical results from the sampling event. Possible staging locations were discussed including one near Building 112 or in Building 167 (suggested by Mr. DeLong), and an alternative location at Building 41 (suggested by Mr. Moss).

Site access will be coordinated through Mr. DeLong and other ROICC personnel, as notice must be given to building tenants at least 24 hours prior to entry. The coordination of access will be conducted by the Navy PM through Mr. DeLong and the City of Alameda. MMEC Group is encouraged to discuss the process currently in-place with SES-TECH for continuity and ease of use.

Radiological areas, such as IR Sites 1 and 32, require that any investigation-derived waste (IDW) from those areas be stored within the areas until cleared by RASO. The IDW must be

SAP WORKSHEET #9 CONTINUED

physically screened out of the areas by a radiological technician. The MMEC Group currently provides radiological support under a separate delivery order for which Mr. Moss is the PM, and support and coordination should be made through him.

Soil gas monitoring is conducted at 9 wells annually at the landfill at IR Site 2, and will be conducted during the spring events. The first event that soil gas will be monitored by MMEC Group will be in spring 2016.

Well decommissioning will take place under this delivery order, and is currently identified in the contract as “up to 10 wells,” although additional wells may be added via a contract modification. Wells will be identified for decommissioning through optimization recommendations, site closure, documented irreparable damage, and identification of wells that are no longer in use.

The 2015 Annual Groundwater Monitoring reporting schedule will remain similar to the current schedule, and will encompass summer and fall 2014 events, as well as winter and spring 2015 events. The MMEC Group proposes to “push up” the reporting dates by approximately 1 month, which the Navy agreed to. It was suggested that the MMEC Group have a face-to-face meeting with SES-TECH and Navy representatives to discuss the planned optimization recommendations that will be presented in the 2014 Annual Groundwater Report, including all of the sampling for the 2014 report being performed by one contractor while the reporting can be performed by a second contractor; this is for consistency in data and reporting. The MMEC Group will use the 2014 report as its template for the 2015 report, utilizing 1-inch binders (or smaller) with larger appendices being included on CDs. For NIRIS submittals, the data are to be submitted by the MMEC Group semi-annually. Mr Sevcik provides Basewide Groundwater Monitoring Program (BGMP) data to other RPMs on a quarterly basis, and requested that the MMEC Group provide him with BGMP data (only lab results) in PDF format, on a quarterly basis.

Source files such as geographic information system (GIS), computer-aided design (dynamic) (CADD), and other files will be requested from SES-TECH by Mr. Olness, as these files are needed in order to produce a Work Plan, SAP, and the Annual Basewide Groundwater Monitoring Report. Mr. Sevcik indicated that this should not be a problem, and recommended that the request be made during the face-to-face meeting with SES-TECH to discuss sampling and reporting requirements, optimization recommendations, and timing of report submittals. Mr. Sevcik indicated that he keeps an electronic copy of IR sites and wells in the Long-term Monitoring Program and that he updates it quarterly, and can provide that list to Mr. Olness.

A technical meeting between the MMEC Group, Navy, and SES-TECH will be scheduled to address the items mentioned above. No further questions were discussed.

The meeting adjourned at approximately 11:15.

SAP WORKSHEET #9 CONTINUED

Project Name: Basewide Groundwater Monitoring Program	**Site Name: Installation Restoration Sites**
Projected Date(s) of Sampling: August 2015 – August 2016	**Site Location: Alameda Point, Alameda, California**
BRAC, NAVFAC SW RPM: Bob Sevcik	

Date of Session: Wednesday, December 17, 2014 at 1330
Scoping Session Purpose: Basewide Groundwater Monitoring Technical Meeting

Name	Title/Role	Affiliation	Phone #	E-mail Address
Bill McGinnis	Lead RPM	NAVFAC SW	(619) 532-0907	william.mcginnis1@navy.mil
Bob Sevcik	RPM	NAVFAC SW	(619) 532-0902	robert.sevcik.ctr@navy.mil
Kevin Olness	Project Manager	MMEC Group	(619) 204-8195	kevin.olness@amecfw.com
Curtis Moss	Technical Support	MMEC Group	(858) 869-7548	curtis.moss@amecfw.com
Lansana Coulibaly	Technical Lead	MMEC Group	(858) 514-6433	lansana.coulibaly@amecfw.com

The meeting began with a discussion on the implementation of optimization recommendations provided in the Annual Reports. Mr. Sevcik and Mr. McGinnis stated that optimization measures would be implemented at the direction of the Navy, prior to the delivery of the Annual Reports if necessary. If an optimization measure required input from the BRAC Closure Team (BCT), then it would be presented at a BCT meeting or wait until the approval of the Annual Report.

The next topic covered were the analytical methods to include in the SAP. It was decided that the list of analytical methods used in the 2013 and 2014 sampling years, as well as the original analytical methods presented in the 2010 SAP would be included in the new SAP to allow for flexibility within the BGMP if needed, even though all analytical methods may not be used throughout the implementation of this BGMP task order.

A review of the IR sites at Alameda Point was conducted by the team next. This review included a brief summary of the current state of each IR site by Mr. McGinnis, discussion on past inclusion/exclusion of each IR site in the BGMP, and potential future activities at each IR site that may or may not exclude the site from the BGMP. Mr. McGinnis stated that if a remedial action was being implemented at an IR site by a separate contractor, that contractor would be responsible for the groundwater issues/monitoring at the site outside of the framework of the BGMP. The IR site may or may not return to the BGMP after completion of the remedial action, pending potential NFA or site closure. It was also stated that wells may be decommissioned at any IR site as needed by the Navy under the framework of the BGMP.

The next topic was a discussion on potential optimization ideas for the BGMP. It was determined a decision tree would be included in the new SAP outlining the decision criteria that would allow for the optimization of the BGMP (target analyte list reduction, monitoring well network reduction, etc.). A statistical analysis of historical and current groundwater data will be used to also assist in the optimization analysis. Mr. Sevcik and Mr. McGinnis stated optimization

SAP WORKSHEET #9 CONTINUED

would be on-going throughout the duration of the BGMP task order and as new ideas arose they would be discussed with the BGMP team as necessary.

The field sampling schedule was covered next by the team. It was stated the first round of sampling for the BGMP required by MMEC Group would be the Fall 2015 (approximately August 2015) sampling event. The sampling events will be conducted quarterly afterward, correlating with the Winter 2015/2016 (approximately December 2015), Spring 2016 (approximately March 2016), and Summer 2016 (approximately May and June 2016) sampling events. Mr. McGinnis stated these quarterly events should be implemented on a streamlined schedule when appropriate. An updated SAP table will be produced prior to each quarterly sampling event documenting the monitoring wells to be sampled that event.

The next topic covered was data requests by MMEC Group to the Navy. MMEC Group requested the following items: a data dump from NIRIS for groundwater data at Alameda Point to allow for MMEC Group to have access to all groundwater data in support of building a database for the BGMP and future reporting activities; a list of all monitoring wells at Alameda Point that may have the potential to be in the BGMP; and source files from previous years Annual Reports to allow for continuity in reporting. The Navy agreed to provide MMEC Group will all requested materials.

The meeting adjourned after the discussion of data requests

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SAP WORKSHEET #10 PROBLEM DEFINITION

This project-specific SAP is prepared following the UFP-QAPP requirements (U.S. EPA, 2005), and is intended for a basewide groundwater monitoring program used to supplement ongoing remedial action and monitored natural attenuation at Alameda Point (Figure A-1).

This worksheet provides the first of seven steps of the DQO process as detailed by the U.S. EPA (U.S. EPA, 2006). Steps 2 through 7 are presented in WS #11. The DQO process is designed to define the project objectives and to ensure that the quality of data collected supports the objectives. This worksheet summarizes the environmental problem to be addressed by the project, the site background, and current conditions to define the key components of the conceptual site model (CSM) for the site.

Site Background

The former Naval Air Station at Alameda (now referred to as Alameda Point or the site) is located on the western end of the island of Alameda (Figure A-1). The site encompasses 2,634 acres, which consists of approximately 1,600 acres of dry land and 1,000 acres of submerged tideland. Alameda Point has been owned and operated by the DoD since 1930 and has served as a naval aircraft maintenance, repair, and refit center. It was also used as a base of operation for naval surface craft (Shaw 2004a, b). Upon closure in April 1997, the site was renamed Alameda Point in a reorientation of the facility toward civilian use. In July 1999, Alameda Point was identified as a National Priorities List (NPL) site and the Navy is currently conducting a site investigation in accordance with CERCLA.

Before 1850, the site was a much smaller peninsula consisting of dry land and marshland. The “island” of Alameda was created in 1902 when dredging of the San Leandro Channel was completed (Bechtel, 2008c). As a result of fill projects conducted between 1871 and 1961, Alameda Island is now three times its original size. Only the eastern portion of Alameda Point consisted of original, dry land. The majority of the site is located on fill up to 40 feet thick (Bechtel, 2008c). From the late 1800s until the 1920s, two manufactured gas plants (MGP) and an oil refinery located near present-day Alameda Point apparently discharged petroleum products and other wastes to adjacent marshlands. These wastes then spread through tidal action over the marsh surface and tidal flats. As the marshlands and subtidal areas were filled, the discharged petroleum products became entrapped in the subsurface, creating what is now referred to as the Marsh Crust and subtidal area (Bechtel, 2008c). The Marsh Crust and subtidal area material consists of a thin (up to 6-inch thick) layer of hydrocarbon-contaminated sediment indicative of coal-based MPG-derived waste.

Wastes historically disposed of at the site include solvents, waste oil, paints, scrap metal, garbage, radiological material, aircraft engines, and possibly munitions and explosives of concern (MEC). In addition, it is estimated that 365,000 gallons of aviation fuel have leaked from underground fuel tanks (ITSI, 2008). Numerous plating and painting shops were also investigated to determine the extent of contamination resulting from past spills and other disposal practices.

The Alameda Point BGMP provides groundwater and soil gas analytical data to be used, in conjunction with other information generated by ongoing site investigation and remedial actions, to define the nature and extent of contamination in groundwater at Alameda Point (ITSI, 2008). A number of individual or commingled groundwater plumes have been identified at Alameda Point.

SAP WORKSHEET #10 CONTINUED

The groundwater plumes generally consist of chlorinated constituents, petroleum hydrocarbons and other fuel-related compounds, metals, or a combination of these contaminants (ITSI, 2008).

Various IR sites have been defined at Alameda Point, some of which are subject to BGMP activities. In some cases, multiple IR sites were grouped into seven OUs (OU-1, OU-2A, OU-2B, OU-2C, OU-3, OU-4A, and OU-6) as shown on Figure A-2. Groundwater sampling and/or soil gas monitoring was conducted following methods and procedures described in previous SAP documents (Shaw 2004b, c; ITSI, 2004; AMEC, 2009, 2010; SES-TECH, 2010, 2011, 2012, 2014). Since the time of the original SAP, conditions at the various IR sites have changed, or in some cases, IR sites have been removed from the BGMP.

The seven Alameda Point OUs are shown on Figure A-2. The 24 sites at Alameda Point that have the potential to be samples as part of this BGMP and overlap the OUs are also shown Figure A-2. Wells associated with the IR sites are shown on Figure A-3 and wells sampled during the most recently completed year of the BGMP, 2014, are shown on Figure A-4 (SES TECH, 2015).

A brief discussion on the current status of the OUs and IR sites, including the most recent trends in groundwater contamination concentrations from the 2014 BGMP sampling results (SES Tech, 2014), as well as references to the Conceptual Site Models (CSMs), are included below.

OU-1

OU-1 originally included IR Sites 6, 7, 8, and 16 in 2007, but has since incorporated IR Sites 14 and 15. Cleanup activities at IR Sites 7, 8, and 15 have been completed. Remedial Actions and/or investigation are on-going at IR Sites 6, 14, and 16 in OU-1 (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015).

OU-1, IR Site 6

IR Site 6 occupies approximately 5.6 acres and includes Building 41, also known as the Aircraft Intermediate Maintenance Facility. The site is generally flat and almost entirely covered with asphalt and concrete in the form of buildings, roads, and parking lots. Chemical contamination at IR Site 6 includes chlorinated solvents such as tricholoroethylene and its byproducts, in soil and groundwater, which are likely related to solvents used in the former washdown areas. This site is currently undergoing remediation in the form of in-situ chemical oxidation and in-situ bioremediation for groundwater contaminants (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015).

The CSM for OU-1, IR Site 6 can be found in the 2014 Final Work Plan for Enhanced In-Situ Bioremediation Groundwater Treatment (MMEC Group, 2014a) or in the Final Record of Decision for Operable Unit 1, Installation Restoration Sites 6, 7, 8, & 16 (BRAC PMO West, 2007b).

OU-1 IR Site 7

IR Site 7 encompasses approximately 5.6 acres and contains Building 549, the Navy Exchange Service Station, and was historically used as an automotive facility. The area was also the site of an incinerator housed in Building 68-3, as well as a craft/hobby shop, laundry facility, maintenance building, and underground storage tanks. Petroleum-related contaminants, PCBs, and metals have been detected in the soil at IR Site 7. Contaminants identified in groundwater at the site include petroleum-related contaminants, sporadic detections of VOCs, and thallium.

SAP WORKSHEET #10 CONTINUED

This site has completed Remedial Action for soil impacts through excavation and removal of approximately 3,400 cubic yards of soil (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015). Groundwater is being remediated under the Alameda Point TPH Program (BRAC, 2007b).

The CSM for OU-1, IR Site 7 can be found in the Final Remedial Action Completion Report, Operable Unit 1, Installation Restoration Site 7 (BRAC PMO West, 2013) or in the Final Record of Decision for Operable Unit 1, Installation Restoration Sites 6, 7, 8, & 16 (BRAC PMO West, 2007b).

OU-1, IR Site 8

IR Site 8 encompasses approximately 4.3 acres and contains Building 114, the former pesticide storage area. Approximately 80% of the site is covered with asphalt and concrete. Building 114 was constructed in 1944 and was operated by the Public Works as the center for weed and pest control. Pesticides, PCBs and VOCS have been detected at low concentrations in the soil at Site 8. Benzene and trichloroethylene have been detected in the groundwater at IR Site 8. Remedial action has been completed at the site via excavation and removal of impacted soil (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015). Groundwater at this IR site is currently being remediated under the Alameda Point TPH Program (BRAC PMO West, 2007b).

The CSM for OU-1, IR Site 8 can be found in the Final Record of Decision for Operable Unit 1, Installation Restoration Sites 6, 7, 8, & 16 (BRAC PMO West, 2007b).

OU-1, IR Site 14

IR Site 14 is the former Fire Fighting Training Area at Alameda Point. IR Site 14 occupies approximately 14.4 acres and was historically used for training firefighters, parking equipment, storing miscellaneous items, defueling planes, cleaning machinery, storing ordinance, maintaining vehicles, storing fuel, and storing and using solvents. The site is partially paved with a generally flat topography (BRAC PMO West, 2007a). IR Site 14 is undergoing remedial action for groundwater in the form of active remediation (in-situ chemical oxidation) combined with monitoring and temporary institutional controls.

The CSM for OU-1, IR Site 14 can be found in the Final Remedial Design/Remedial Action Work Plan (Battelle and ITSI, December 2008) and in the Final Record of Decision for Installation Restoration Site 14, Former Firefighting Training Area (BRAC PMO West, 2007).

Trends in Groundwater Contamination Concentrations

Groundwater contamination trends in the first water bearing zone (FWBZ) at IR Site 14 were evaluated for vinyl chloride (VC) because of its history of detections at or above the Institutional Control Termination Criterion (ICTC).

VC concentrations are increasing in one well (M14-09D), decreasing in three wells (M14-09S, M14-14, and M14-16), and relatively stable, on average, in the remaining four wells, although M14-06 and M14-15 show high variability. The average VC trend concentrations exceed the ICTC of 15 µg/L in five of the eight wells for which trends were evaluated (SESTech, 2014). Groundwater conditions IR Site 14 will be monitored as part of the BGMP until the ICTC are met per the ROD.

SAP WORKSHEET #10 CONTINUED

OU-1, IR Site 15

IR Site 15 encompasses approximately 6 acres and includes Buildings 301, 389, and 27. Most of the site is unpaved, including approximately 1 acre of wetlands in the northern border of the site near the Oakland Inner Harbor. Building 27 is the only building that remains on the site. The site was primarily used for the storage of petroleum products, biocides, electrical equipment, oil-filled transformers, and machinery. Contamination included PCBs (Aroclor 1260) and lead, which may have been released to soil from spills or leaking oil-containing transformers, use of PCB-containing oil for weed control, or storage and use of lead-based paints (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015).

IR Site 15 has been determined not to pose an unacceptable risk to human health or the environment, and accordingly, has been recommended for no further action for groundwater in the 2006 Final Record of Decision.

The CSM for OU-1, IR Site 15 can be found in the Final Record of Decision, Former Transformer Storage Area, Site 15 (SulTech, 2006a).

OU-1, IR Site 16

IR Site 16 encompasses approximately 11.1 acres in the southeastern corner of Alameda Point. Site 16 is known as the C-2 CANS Area, or the Shipping Container Storage Area. Eight groups of shipping containers have been used to store avionic parts and test equipment, as well as solvents. IR Site 16 was also used for aircraft parking and the storage of paints, solvents, acids and bases, as well as transformers containing PCB oil (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015). This site is undergoing remediation in the form of in-situ chemical oxidation and in-situ bioremediation for groundwater contaminants (BRAC PMO West, 2007b). A ROD explanation of significant difference (ESD) is currently being prepared to revise the RAOs for groundwater at IR Site 16 (MMEC Group, 2014b). The ROD ESD is anticipated to be finalized in late 2015.

The CSM for OU-1, IR Site 16 can be found in the Final Record of Decision for Operable Unit 1, Installation Restoration Sites 6, 7, 8, and 16 (BRAC PMO West, 2007b) and the Draft ROD ESD (MMEC Group, 2014b).

OU-2A

OU-2A originally included IR Sites 9, 13, 19, 22, and 23. OU-2A encompasses approximately 39.5 acres east of the Seaplane Lagoon and west of Central Avenue in the southeastern portion of Alameda Point. Cleanup activities at IR Sites 9, 19, 22, and 23 have been completed. Only the remedial action at IR Site 13 in OU-2A is ongoing.

OU-2A, IR Site 9

IR Site 9 encompasses approximately 2.9 acres in the southwestern corner of OU-2A. There are currently two buildings on the site; Building 410 and Building 351. Building 410 was constructed in 1988 and was used as an aircraft paint stripping facility, and used for storage since the early 1990s. Building 351, located to the north of Building 410 served as support for Building 410, and housed lunch and locker rooms (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015). IR Site 9 will employ institutional controls restricting the use of groundwater until remediation goals are met (BRAC PMO West, 2012).

SAP WORKSHEET #10 CONTINUED

The CSM for OU-2A, IR Site 9 can be found in the Final Record of Decision for OU-2A (BRAC PMO West, 2012).

OU-2A, IR Site 13

IR Site 13 in OU-2A is the former Oil Refinery area at Alameda Point. IR Site 13 encompasses approximately 17.5 acres, is partially paved, and has generally flat topography. The site was the location of the Pacific Coast Oil Works Company from 1879 to 1903 (BRAC PMO West, 2012). IR Site 13 is undergoing in-situ bioremediation, monitored natural attenuation, and employs institutional controls requiring the installation of an approved vapor control system for any new residential construction until vapor intrusion remediation goals are met and restricting the use of groundwater (BRAC PMO West, 2012).

The CSM for IR Site 13 can be found in the Final Record of Decision OU-2A (BRAC PMO West, 2012) or in the Final Feasibility Study Report, Operable Unit 2A, Sites 9, 13, 19, 22, and 23 (OTIE, 2011b).

Trends in Groundwater Contamination

Groundwater contamination concentration trends for IR Site 13 were evaluated for one well (S13-TT-MW01) in the FWBZ during the Winter and Spring 2014 sampling events. Benzene and ethylbenzene concentration trends appear to be stable below their RGs at that location (SESTech, 2014).

OU-2A, IR Site 19

IR Site 19 encompasses approximately 2.3 acres in the northwestern corner of OU-2A. Currently there are two buildings within IR Site 19; Building 616 and Yard D-13. Building 616 was constructed in 1982 and was used for office and storage space. Yard D-13 was a RCRA-permitted hazardous waste storage area. Yard D-13 stopped accepting wastes in 1996 and received closure from the DTSC in 1999. IR Site 19 previously contained Building 609, which was demolished in 1991, but was constructed in 1975 and was used for building and storing engines (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015). IR Site 19 will employ institutional controls until remediation goals are met. There are Petroleum Program activities within IR Site 19 (BRAC PMO West, 2012).

The CSM for OU-2A, IR Site 19 can be found in the Final Record of Decision OU-2A (BRAC PMO West, 2012).

OU-2A, IR Site 22

IR Site 22 encompasses approximately 2.1 acres in the northeastern corner of OU-2A. The site was previously used for barracks until the 1970's, when a service station was constructed. All buildings associated with the service station have been demolished, although some foundations still exist at the site (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015). No action is required at IR Site 22 (BRAC PMO West, 2012).

The CSM for OU-2A, IR Site 22 can be found in the Final Record of Decision OU-2A (BRAC PMO West, 2012).

SAP WORKSHEET #10 CONTINUED

OU-2A, IR Site 23
IR Site 23 encompasses approximately 14 acres in the southern portion of OU-2A. Historically, the Pacific Coast Oil Works Company petroleum refinery operated within the site from 1879 until 1903. Between 1953 and the early 1970's, portions of the site were uses for airplane defueling activities. The main structure was Building 530, which was constructed in 1973 for missile re-work operations. Operational support functions were provided at two adjacent buildings (529 and 600). Currently, the site is used for vehicle storage and parking (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015). No action is required at IR Site 23, though there are Petroleum Program activities taking place within IR Site 23 (BRAC PMO West, 2012).

The CSM for OU-2A, IR Site 23 can be found in the Final Record of Decision OU-2A (BRAC PMO West, 2012).

OU-2B
OU-2B encompasses approximately 46.6 acres on the eastern portion of the base, and was used for industrial proposes in the late 1800's by the Pacific Coast Oil Company. The Navy acquired the land in 1936, and the base was constructed several iterations of the tidelands and marshlands. Areas of OU-2B were filled from 1942-1946. OU-2B consists of four CERCLA sites: IR Site 3 (abandoned fuel storage area), IR Site 4 (aircraft engine test facility [Building 360]), IR Site 11 (engine test cell [Building 14]), and IR Site 21 (ship fitting and engine repair [Building 162]).

OU-2B, IR Site 3
IR Site 3, also known as the Abandoned Fuel Storage Area, covers approximately 13.4 acres near the eastern end of Alameda Point, between West Atlantic Avenue and West Trident Avenue. An estimated 75% of the site is covered with asphalt and concrete in the form of buildings, roads, and parking lots, with the remaining 25% of the site being landscaped. From the 1940's to the 1970s, aviation gasoline was stored in underground storage tanks at the site. The OU-2B shallow groundwater plume does not extend beneath IR Site 3, but because the buffer area for the plume extends across the southern boundary of IR Site 3, institutional controls for OU-2B shallow groundwater apply to that portion of IR Site 3 (BRAC PMO West, 2015).

The CSM for OU-2B, IR Site 3 can be found in the Final Record of Decision, Operable Unit 2B (BRAC PMO West, 2015) or in the Final Feasibility Study Report, Operable Unit 2B, Installation Restoration Sites 3, 4, 11, and 21 (OTIE, 2011c).

Trends in Groundwater Contamination
Groundwater contamination trends for IR Site 3 were evaluated for dissolved metals (arsenic and iron) because both arsenic and iron concentrations exceed their respective BVs. The trends of dissolved arsenic and dissolved iron in groundwater appear to indicate stable to slightly declining concentrations over time (SESTech, 2014).

OU-2B, IR Site 4
IR Site 4 is Building 360, also known as the Aircraft Engine Facility at Alameda Point. IR Site 4 covers approximately 22.7 acres near the eastern end of Alameda Point south of West Atlantic Avenue. An estimated two-thirds of the site is covered with asphalt and concrete in the form of buildings, roads, and parking lots, with the remaining one-third being landscaped. IR Site 4

SAP WORKSHEET #10 CONTINUED

includes Building 360, which was used for aircraft engine and airframe overhaul. VOCs in shallow groundwater beneath IR Site 4 will be addressed using in-situ bioremediation, monitoring, and institutional controls (BRAC PMO West, 2015).

The CSM for OU-2B, IR Site 4 can be found in the Final Record of Decision, Operable Unit 2B (BRAC PMO West, 2015) or in the Final Feasibility Study Report, Operable Unit 2B, Installation Restoration Sites 3, 4, 11, and 21 (OTIE, 2011c).

Trends in Groundwater Contamination
Groundwater contamination concentration trends in the FWBZ and second water bearing zone (SWBZ) were evaluated for TCE and VC because of their widespread presence at IR Site 4 and their history of detections at or above RGs.

In the FWBZ, VC concentration trends are declining in wells S4-TT-MW02A, MW360-4, and M03-10. In the SWBZ, wells S4-TT-MW02C and S4-TT-MW02E show decreasing TCE trends since 2010, and well S4-TT-MW03B shows a fluctuating, but generally declining TCE trend. All other wells evaluated for TCE exhibit a generally stable trend for TCE.

In the FWBZ, VC concentration trends are declining or stable below the RG in wells S4-TT-MW02A, MW360-4, and M03-10. Wells S4-TT-MW03A and MW4-2-1 show increasing VC trends. In the SWBZ, well S4-TT-MW02B shows a declining VC trend since Winter 2012. Wells S4-TT-MW02C, S4-TT-MW03B, and S4-TT-MW03C show increasing VC trends. All other wells evaluated for VC exhibit generally stable trend for VC (SESTech, 2014).

OU-2B, IR Site 11
IR Site 11 in OU2B is Building 14, the Engine Test Cell at Alameda Point. IR Site 11 covers approximately 5.4 acres near the eastern edge of Seaplane Lagoon between Ferry Point Road and Orion Street. Nearly all of the site is covered with asphalt and concrete in the form of buildings, roads, and parking lots, with minimal landscaping. IR Site 11 includes Building 14, and engine test cell constructed in 1940 and operated as an aircraft testing and repair facility. VOCs in shallow groundwater beneath IR Site 11 will be addressed using in-situ bioremediation, monitoring, and institutional controls (BRAC PMO West, 2015).

The CSM for OU-2B, IR Site 11 can be found in the Final Record of Decision, Operable Unit 2B (BRAC PMO West, 2015) or in the Final Feasibility Study Report, Operable Unit 2B, Installation Restoration Sites 3, 4, 11, and 21 (OTIE, 2011c).

Trends in Groundwater Contamination
Groundwater contamination concentration trends in the FWBZ and SWBZ were evaluated for TCE and VC because these COCs exceeded their RGs. In the FWBZ, TCE concentrations in wells S11-TT-MW04A and S11-TT-MW01A are declining. In the SWBZ, at well S11-TT-MW03B, TCE concentrations have been stable, without significant upward or downward trends. At well S11-TT-MW01B, a decreasing trend is apparent. At well S11-TT-MW03C, a weak average decline is discernible, although this trend is highly erratic. At well S11-TT-MW04B, an increasing trend is apparent. VC concentrations in the SWBZ have been stable, without significant increasing or decreasing trends in wells S11-TT-MW03B and S11-TT-MW03C. At well S11-TT-MW01B, a decreasing VC concentration trend is apparent. At well S11-TT-MW04B, an increasing VC concentration trend is apparent (SESTech, 2014).

SAP WORKSHEET #10 CONTINUED

OU-2B, IR Site 21

IR Site 21 in OU-2B is Building 162, the Ship Fitting and Engine Repair facility at Alameda Point. IR Site 21 covers approximately 5.1 acres near the eastern edge of Seaplane Lagoon south of West Atlantic Avenue. About half of the site is covered with asphalt and concrete in the form of buildings, roads, and parking lots, with the remaining half of the site being landscaped or exposed ground. IR Site 21 includes Building 162, which was constructed in 1945 as a ship and aircraft maintenance shop. VOCs in shallow groundwater beneath IR Site 21 will be addressed using in-situ bioremediation, monitoring, and institutional controls (BRAC PMO West, 2013).

The CSM for OU-2B, IR Site 21 can be found in the Final Record of Decision, Operable Unit 2B (BRAC PMO West, 2015) or in the Final Feasibility Study Report, Operable Unit 2B, Installation Restoration Sites 3, 4, 11, and 21 (OTIE, 2011c).

Trends in Groundwater Contamination

Groundwater contamination concentration trends in the FWBZ at IR Site 21 were evaluated for TCE; cis-1,2-DCE; and VC because of their history of detections at or above the RGs. In the FWBZ, cis-1,2-DCE concentrations show a generally stable trend below the RG since 2008 until the fall of 2013 sampling event, when the cis-1,2-DCE concentrations in one well spiked above the RG before returning to detection limit in Spring 2014. The TCE concentrations show an erratic, but generally declining trend in well M11-06, and VC concentrations show a generally increasing trend in the same well. In the SWBZ, the TCE concentrations show erratic but generally declining trends in both wells S21-TT-MW05D and S121-TT-MW05E. VC concentrations show generally stable trends in wells S21-TT-MW05D and S21-TT-MW05E (SESTech, 2014).

OU-2C

OU-2C encompasses approximately 53 acres and is located in the middle portion of Alameda Point and contains IR Site 5 (the former Naval Air Rework Facility), IR Site 10 (the former missile rework facility), and IR Site 12 (including Building 10, a former power plant). OU-2C encompasses approximately 53 acres in the middle of Alameda Point north of Seaplane Lagoon and west of the former runways and hangars.

OU-2C, IR Site 5

IR Site 5 in OU-2C is the Naval Air Rework Facility and contains the Building 5 Area (approximately 20.9 acres) at Alameda Point. IR Site 5 covers approximately 46.9 acres north of Seaplane Lagoon on the northwestern corner of the intersection of West Tower Avenue and Monarch Street at Alameda Point. Nearly all of the site is covered with asphalt and concrete in the form of buildings, roads, and parking lots, with the remainder of the site being landscaped or exposed ground. IR Site 5 includes Building 5, which was used as an aircraft rebuilding facility (Tetra Tech, 2012a). IR Site 5 currently implements in-situ chemical oxidation, enhanced bioremediation, groundwater monitoring, and institutional controls for FWBZ groundwater because of elevated concentrations of VOCs. Institutional controls have been declared for FWBZ and SWBZ because of elevated concentrations of VOCs (BRAC PMO West, 2014). A groundwater treatment is being evaluated and may be implemented at IR Site 5 in the next year at which time groundwater monitoring will no longer be implemented under the BGMP.

SAP WORKSHEET #10 CONTINUED

The CSM for OU-2C, IR Site 5 can be found in the Final Record of Decision OU-2C (IR Sites 5, 10, and 12), or in the Final Addendum 1 to the Operable Unit 2C Feasibility Study Report, IR Sites 5 and 10 (Tetra Tech, 2012a).

Trends in Groundwater Contamination

Groundwater contamination concentration trends in the FWBZ were evaluated for 1,1-DCA, TCE, and VC because VOCs have RGs. No significant trends were identified for the FWBZ because results continue to be below RGs. In the FWBZ, 1,1-DCA concentrations, although locally variable, have remained relatively stable, with no significant increasing or decreasing trends in three (M05-02, M05-09, and M05-HW01) of the seven wells evaluated. At well M05-15, 1,1-DCA concentrations have been stable since a one order-of-magnitude decrease in Spring 2011. The 1,1-DCA concentrations in well 2MW8S are declining. In well M05-05, 1,1-DCA concentrations continue to decline after an order-of-magnitude decrease in Fall 2009. At well M05-20, 1,1-DCA concentrations show an order of magnitude decrease from a spike in concentrations observed in Spring 2011. Well M05-16 shows a slightly increasing to stable trend in 1,1-DCA. TCE results for wells M05HW-01, 2MW8S, M05-05, and M05-20 all show increases, although all TCE concentrations remain significantly below the RG. TCE concentrations are declining in wells M05-02, M05-15, and possibly M05-16. VC concentrations appear to have increasing trends in three (M05-02, M05-16, and M05HW-01) of the seven wells evaluated, and decreasing trends in three (M05-05, M05-20, and 2MW8S) of the seven wells evaluated. At well M05-15, VC has a declining trend. At well M05-15, VC concentrations have remained stable at the reporting limit since May 2011 (SESTech, 2014).

OU-2C, IR Site 10

IR Site 10 encompasses approximately 4 acres and was the former missile rework facility. This IR Site is bounded on the north by IR Site 5, and is approximately 600 feet north of Seaplane Lagoon. Building 400 occupies approximately 85% of the site, and the remaining 15% is made up of paved open space (i.e., parking lots and roads). Past uses of IR Site 10 include paint stripping, construction of fiberglass airplane components, airplane parts cleaning and degreasing, silk screening, and photographic development. The radium paint shop facilities for the painting of radioluminescent (^{226}Ra) aircraft dials were moved from Building 5 to Building 400 in the late 1950's. IR Site 10 is currently in the remedial design phase, and is not currently part of the BGMP (BRAC PMO West, 2014).

OU-2C, IR Site 12

IR Site 12 encompasses approximately 2 acres and includes Building 10, unpaved areas, and roads and parking lots. Building 10 was constructed in 1940 as a power plant, and operated until base closure in 1997. Historical activities included steam generation and air compression. IR Site 12 is currently in the remedial design phase, and is not currently part of the BGMP (BRAC PMO West, 2014).

OU-3

OU-3 encompasses approximately 96 acres in the northwestern corner of Alameda Point where the Oakland Inner Harbor joins the San Francisco Bay, and includes IR Sites 1 and 32. The area was historically used to dispose of waste, store aircraft parts and petroleum, and as a pistol and skeet range. The area is partially paved and has a relatively flat topography with slight depressions (Envirostor – www.envirostor.dtsc.gov – accessed 5/29/2015).

SAP WORKSHEET #10 CONTINUED

OU-3, IR Site 1 and IR Site 32

IR Site 1 is the 1943-1956 Disposal Area, and associated IR Site 32 is the Northwest Ordinance Storage Area at Alameda Point. IR Site 1 was formerly used for waste disposal, a skeet/pistol range, and aircraft parts storage. Most of the site is exposed ground, with a minor portion covered with asphalt and concrete in the form of roads, runways, and tarmac (ChaduxTt, 2009). According to the ROD developed and approved in 2009, VOCs inside the plume area in IR Site 1 will be addressed via in-situ chemical oxidation, or by a similar process. The plume area will be monitored to determine the effectiveness of treatment. Institutional controls will also be in place to prohibit activities that may reduce the effectiveness of the remedy, and that prohibit the construction of buildings over the VOC plume, unless approved by the FFA signatories (ChaduxTt, 2009). The remedial action at IR Sites 1 and 32 is currently on-going and after the completion of the remedial actions groundwater monitoring activities at the sites will be continued under the BGMP.

The CSM for OU-3, IR Site 1 is presented in the Final Record of Decision for Installation Restoration Site 1, 1943-1956 Disposal Area (ChaduxTt, 2009) and the CSM for OU-3, IR Site 32 is presented in the Final Feasibility Study Report, IR Site 32, Northwestern Ordnance Storage Area (Bechtel, 2008a).

Trends in Groundwater Contaminant Concentrations

Groundwater contamination concentration trends in the FWBZ at IR Site 1 were evaluated for benzene, 1,1-DCE, TCE, VC, and radionuclides. Benzene concentrations have shown no significant overall trend in two of the four wells evaluated (M033-A and M034-A). Wells M029-A and M028-E exhibit overall slight declining trends in benzene, with significant fluctuation, since 2007. 1,1-DCE concentrations show no significant trend in three of the four wells evaluated. An increase has been observed in 1,1-DCE concentrations at well M034-A, beginning in Spring 2013. TCE concentrations show no significant trend in three of the four wells evaluated. A recent increase in TCE concentration was observed at M034-A beginning in Spring 2013. At well M028-E, after some significant variability in concentrations until mid-2009, TCE concentrations appear to have become stable at 1.0 ug/L or less. VC concentrations have shown no significant trend in three of the four wells evaluated. A general upward trend (with significant fluctuation) continues to be observed at well M034-A. VC concentrations in well M028-E show significant variability (three orders of magnitude) since 2007, but in general appear to have a declining trend. Gross alpha activity is stable near 0 pCi/L, and gross beta activity is also generally stable, with occasional temporary spikes in well M034-A. In well M026-A, both gross alpha and gross beta activity appears to fluctuate similarly, within an absolute value of 250 pCi/L or less in the wells evaluated. No increasing or decreasing trends were observed (SESTech, 2014).

OU-4A

OU-4A is the West beach landfill and Wetlands at Alameda Point. The VA FED parcel is generally described as the area east of IR Site 2, bounded by former runway No. 31. OU-4A encompasses approximately 110 acres in the southwestern corner of Alameda Point (BRAC PMO West, 2010).

OU-4A, IR Site 2, and VA FED Parcel

IR Site 2 covers approximately 110 acres at the extreme southwestern corner of Alameda Point. IR Site 2 was formerly used for waste disposal, including the 60-acre West Beach Landfill, and

SAP WORKSHEET #10 CONTINUED

includes wetlands. Most of the site is exposed ground and engineered soil cover, with a minor portion covered with asphalt and concrete in the form of roads, runways, and tarmac (BRAC PMO West, 2010). The VA FED parcel is generally contiguous with the eastern border of IR Site 2, and is bounded by former runway No. 31. About half of the parcel is exposed ground with a minor portion covered by asphalt and concrete in the form of roads and tarmac (BRAC PMO West, 2010). The remedy selected in the 2010 ROD for groundwater at IR Site 2 includes monitored natural attenuation, implementing engineering controls and institutional controls to protect human health and the groundwater remedy (BRAC PMO West, 2010). The remedial action at IR Site 2 is complete as of mid-2015 and long term groundwater monitoring of the site will be conducted under the BGMP.

The CSM for OU-4A, IR Site 2 can be found in the Final Record of Decision, IR Site 2 (BRAC PMO West, 2010) and in the Final Feasibility Study Report, IR Site 2, West Beach Landfill and Wetlands (Battelle and BBL, Inc., 2008).

The CSM for VA FED Parcel can be found in the Final Site Inspection Report, Transfer Parcels FED-1A, FED-2B, and FED-2C (CH2MHill, 2011).

Trends in Groundwater Contaminant Concentrations
Groundwater contamination concentration trends in the FWBZ were evaluated for dissolved metals (arsenic, copper, and nickel) at IR Site 2 and the VA FED parcel because these metals have historically exceeded their BVs. Dissolved arsenic results show neither increasing nor decreasing trends, disregarding the artificially elevated arsenic concentrations detected from Spring 2009 to Spring 2010 due to elevated reporting limits during that period. Dissolved copper results also show neither increasing nor decreasing trends, except at two wells, disregarding the artificially elevated copper concentrations detected from Spring 2009 to Spring 2010 due to elevated reporting limits during that period. Wells M020-E and M023-E show and increasing dissolved copper trend since Spring 2012. Dissolved nickel results also show neither an increasing nor decreasing trend except at two wells, disregarding the artificially elevated nickel concentrations detected from Spring 2009 to Spring 2010 sue to elevated reporting limits during that period. Since Spring 2012, well M020-E shows an increasing nickel trend and well M036-E shows a decreasing nickel trend (SESTech, 2014).

OU-5
OU-5 includes IR Sites 25, 30, and 31, as well as Fleet and Industrial Supply Center Oakland, Alameda Facility/Alameda Annexes (FISCAs) IR-01, IR-02, and IR-03. The primary land use for OU-5 is residential, while the land use at FISCA is mixed. In accordance with the RI/FS and ROD, the selected remedy for OU-5/FISCA IR-02 was: biosparging in the plume centers, with soil vapor extraction and nutrient/microorganism enhancement as required, monitored natural attenuation for the remaining plume (and within the plume centers following completion of the active remediation), and institutional controls while the remediation is in progress (Tetra Tech, 2012b). In 2014, the U.S. Navy proposed “No Further Action” for groundwater at the OU-5/FISCA IR-02. A ROD Amendment is forthcoming, currently scheduled for submittal on mid-2015.

IR Site 25
IR Site 25 encompasses approximately 42 acres, consisting of unoccupied North Village Housing and the U.S. Coast Guard Housing Maintenance Office.

SAP WORKSHEET #10 CONTINUED

IR Site 30
IR Site 30 encompasses approximately 6 acres, and consists of Island High School and the Woodstock Child Development Center.

IR Site 31
IR Site 31 encompasses approximately 24 acres, and consists of the U.S. Coast Guard Marina Village Housing Area.

FISCA IR-01
FISCA IR-01 is a former warehouse area located south of IR Site 31, on the southwest side of FISCA, and has been redeveloped into Bayport Housing.

FISCA IR-02
FISCA IR-02 is located on the south central side of FISCA. The Defense Logistics Agency Defense Reutilization and Marketing Office operated a screening lot and scrap yard at IR-02 until 1997. The western portion of IR-02 was used as a screening lot and for temporary equipment storage, while the eastern portion of IR-02 was used as a scrap yard and for temporary storage of discarded automobiles, stockpiled scrap mental, and surplus equipment. The Shinsei garden Housing Development, a multi-family residential project, has been constructed on the western portion of IR-02.

FISCA IR-03
FISCA IR-03 is located in the west central portion of FISCA. It previously consisted of an automotive drive-up maintenance rack over an asphalt paved area. This area remains undeveloped (Tetra Tech, 2012b).

The CSM for OU-5, IR Sites 25, 30, and 31 is presented in the Final Technical Memorandum, Operable Unit 5/FISCA IR-02 Groundwater Data Evaluation, Alameda Point OU-5 (Sites 25, 30, and 31) Groundwater and FISCA Site IR-02 Groundwater (Tetra Tech, 2012b).

OU-6
OU-6 consists of IR Site 26 (Western Hangar Zone), IR Site 27(the Dock Zone), and IR Site 28 (the Todd Shipyard). OU-6 encompasses approximately 47.5 acres in three discontiguous parcels within Alameda Point.

OU-6, IR Site 26
IR Site 26, is the Western Hangar Zone at Alameda Point. IR Site 26 covers approximately 32 acres in the central portion of Alameda Point, immediately east of a partially paved runway area, and west of IR Site 5. IR Site 26 was formerly used for aircraft maintenance. Most of the site is covered with asphalt and concrete in the form of roads, parking lots, and buildings (SulTech, 2006b).

The ROD, submitted in 2006, selected the following remedy for groundwater at IR Site 26: in-situ chemical oxidation, in-situ bioremediation, short-term institutional controls, and sampling of groundwater to confirm the achievement of remediation goals for cis-1,2-dichloroethene, trichloroethene, and VC (SulTech, 2006b).

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The CSM for OU-6, IR Site 26 can be found in the Final Record of Decision, Site 26 (SulTech, 2006b).

Trends in Groundwater Contaminant Concentrations
Groundwater contamination concentration trends in the FWBZ were evaluated for cis-1,2-DCE and VC in wells 26PZ01, 26PZ02, 26PZ03, 26MW03, and 26MW08 at IR Site 26 because of their history of detection about the RGs in these wells. Cis-1,2-DCE concentrations have varied, but the overall trend is declining on average in all wells except well 26PZ02. Although cis-1,2-DCE concentrations in this well are below the RG, the cis-1,2-DCE trend appears generally stable. VC concentrations have been highly variable in some wells, but the overall trend is on average declining in wells 26MW03 and 26MW08, and slightly increasing in wells 26PZ02 and 26PZ03. The VC trend in well 26PZ01 is highly variable, but is on average stable (SESTech 2014).

OU-6, IR Site 27
IR Site 27 in OU-6 is the Dock Zone at Alameda Point. IR Site 27 covers approximately 12.6 acres in the southeastern corner of Alameda Point, immediately east of Seaplane Lagoon. Most of the site is covered with asphalt and concrete in the form of roads, parking lots, and buildings (Bechtel, 2008b).

The CSM for OU-6, IR Site 27 is presented in the Final Record of Decision for Installation Restoration site 27, Dock Zone (Bechtel, 2008b).

Trends in Groundwater Contaminant Concentrations
Groundwater contamination concentration trends were evaluated for cis-11,2-DCE and VC in 20 wells at IR Site 27 because of their history of detections at or above the RGs. Of the 20 wells sampled, 14 wells had sample results exceeding the RG for cis-1,2-DCE, VC, or both. Cis-1,2-DCE and VC did not exhibit discernible trends except for wells 27EW-08, 27EW-18, and 27MW06, which show declining trends for both cis-1,2-DCE and VC. Well 15-MW2 shows a declining trend for cis-1,2-DCE, and well 27EW-12 shows a declining trend for VC. In addition, results for well 27EW-05 indicated increasing trends for both cis-1,2-DCE and VC, and results for well 15-MW1 indicate an increasing trend for VC (SESTech, 2014).

OU-6, IR Site 28
IR Site 28 is the Todd Shipyard at Alameda Point, covers approximately 2.9 acres, is irregular in shape, and is located on the northern edge of Alameda point, immediately south of the Oakland Inner Harbor. IR Site 28 includes a dog park and a partially paved parking lot (Battelle, 2012).

The CSM for OU-6, IR Site 28 can be found in the Final Interim Remedial Action Completion Report, Installation Restoration Site 28 (Battelle, 2012).

Trends in Groundwater Contaminant Concentrations
Groundwater contamination concentration trends were evaluated for copper only, because copper is the only COC detected at concentrations at or above the RG. Copper in well 28SW03 generally shows a declining trend. Copper in wells 28SW02 and 28SW08 previously showed a stable trend until the Winter 2012 monitoring period, when copper concentrations began increasing in both wells. Wells 28SW02 and 28SW08 both show an increasing copper trend (SESTech 2014).

SAP WORKSHEET #10 CONTINUED

Other IR Sites
These IR Sites are not currently or historically associated with an OU at Alameda Point.

IR Site 34
IR Site 34 encompasses approximately 4.2 acres and was once part of the San Francisco Bay along the Oakland Inner Harbor's southern border. During the late 1800s and early 1900s, a Southern Pacific railroad track existed on a narrow strip of filled land across the northern portion of IR Site 34. IR Site 34 consists of EPB Parcels 4, 18, and a small unused portion of EBS Parcel 16, as well as an unnamed portion of land between the EBS parcels and the Oakland Inner Harbor. The Navy constructed 12 buildings at IR Site 34, though all buildings and associated structures were removed between 1996 and 2000. Additional features at the site included seven aboveground storage tanks, three RCRA sites, electrical transformers, and open space (Envirostor – www.envirostor.dtsc.gov, accessed 5/29/2015).

The CSM for IR Site 34 is presented in the Final Remedial Action Completion Report, Installation Restoration Site 34 (ERS, 2014).

IR Site 35
IR Site 35 encompasses approximately 75 acres, and consists of open space areas with no buildings present, residences, and commercial/industrial buildings. IR Site 35 is generally bounded by the Oakland Inner Harbor to the north, Main Street to the east, Transfer Parcel EDC-10 and Seaplane Lagoon to the south, and Transfer Parcels EDC-7, EDC-9, EDC-15, and Transfer Parcel Public Benefit Conveyance (PBC)-1A to the west. Historical uses of IR Site 35 were industrial, residential and recreational. These uses included living quarters, medical facilities, maintenance facilities, water towers, air terminal offices, educational buildings, hobby shops, dog training and kenneling facilities, a plant nursery, material storage tanks, and oil/water separators (OTIE, 2010).

The CSM for IR Site 35 can be found in the Final Record of Decision for Installation Restoration Site 35 Areas of Concern in Transfer Parcel EDC-5 (OTIE, 2010).

A summary of the current status of each OU and IR Site is included in Table 10-1.

SAP WORKSHEET #10 CONTINUED

Table 10-1 Summary of Current OU and IR Site Status for Groundwater

OU	IR Site	Current Status
OU-1	IR Site 6	Active Remediation
	IR Site 7	Active Remediation (Petroleum Program)
	IR Site 8	No Further Action
	IR Site 14	MNA
	IR Site 15	No Further Action
	IR Site 16	MNA
OU-2A	IR Site 9	No Groundwater Monitoring Required
	IR Site 13	MNA + Active Bioremediation (partial site)
	IR Site 19	No Groundwater Monitoring Required
	IR Site 22	No Further Action
	IR Site 23	No Further Action
OU-2B	IR Site 3	No Further Action Pending
	IR Site 4	MNA + Active Bioremediation
	IR Site 11	MNA + Active Bioremediation
	IR Site 21	MNA + Active Bioremediation
OU-2C	IR Site 5	Active Remediation
	IR Site 10	No Further Action
	IR Site 12	No Further Action
OU-3	IR Site 1	Groundwater Release Detection Monitoring
OU-4A	IR Site 2	Groundwater Release Detection Monitoring
OU-5	IR Site 25	No Further Action
	IR Site 30	No Further Action
	IR Site 31	No Further Action
OU-6	IR Site 26	MNA
	IR Site 27	MNA
	IR Site 28	MNA
Other	IR Site 32	Feasibility Study Phase
	IR Site 33	No Further Action
	IR Site 34	No Further Action
	IR Site 35	No Further Action
Notes:		
	* IR Sites 17, 20, 24, and 29 are submerged sites with no groundwater monitoring requirement	
	** IR Site 18 Storm Drain System was merged into other sites and closed.	

SAP WORKSHEET #10 CONTINUED

Topography

Based on well elevation surveys, the surface elevation at Alameda Point is approximately 7 to 17 feet above mean sea level (msl). The site is relatively flat with portions of the facility developed, paved, and partially covered with vegetation. Other areas have been left undeveloped, unpaved, and generally covered with vegetation.

Geology

Alameda Point occupies a depression between two uplifted areas: the Berkeley Hills to the east and the Santa Cruz Mountains on the San Francisco Peninsula to the west. The depression and uplifted areas are formed by two subparallel, active faults: the San Andreas and the Hayward Faults. Alameda Point and the surrounding San Francisco Bay are underlain by 400 to 500 feet of unconsolidated sediments that overlay the metamorphosed sandstone, siltstone, shale, greywacke, and igneous bedrock, which forms the Franciscan Formation (Bechtel, 2008c).

Surface and near-surface soil at Alameda Point consists of artificial fill emplaced during historical filling of the tidal marshlands and the subtidal area of San Francisco Bay during site development. The fill material consists of sediments that were dredged from the tidal flats of San Francisco Bay and Oakland Inner Harbor and is characterized by sands, clays, and silts (Bechtel, 2008c). The unconsolidated sediments that lie beneath the artificial fill consist of the following five units, from top to bottom: (1) the Bay Sediment Unit (BSU), (2) the Merritt Sand Formation, (3) the upper unit of the San Antonio Formation, (4) the lower unit of the San Antonio Formation (Yerba Buena Mud), and (5) the Alameda Formation.

Hydrogeology

Groundwater is present at Alameda Point at approximately 1 to 8 feet below ground surface (bgs) in the artificial fill. From top to bottom, the following five distinct hydrostratigraphic zones (HSZ) have been identified at Alameda Point (although not all occur at all locations):

- First Water Bearing Zone (FWBZ) (Artificial Fill)
- Semi-confining aquitard (BSU/Young Bay Mud)
- Second Water Bearing Zone (SWBZ) (Posey, Merritt Sand, and upper San Antonio Members)
- Yerba Buena Mud Aquitard, a regional aquitard
- Alameda Aquifer

All units are members of the Alameda Formation, except for the artificial fill.

The hydrostratigraphic zones are described as follows:

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- ***First Water Bearing Zone:*** The FWBZ consists of shallow unconfined groundwater in artificial fill, the coarse-grained Bay BSU, and locally, the upper Merritt Sand. The FWBZ is divided into two hydrologically distinct subintervals in some areas: FWBZ-Upper (composed primarily of artificial fill) and FWBZ-Lower (comprised of BSU sands and upper Merritt Sand). Groundwater is first encountered in the FWBZ at depths of approximately 2 to 8 feet bgs. The FWBZ is tidally influenced for approximately 250 to 300 feet inland on the northern and southern sides of the Island of Alameda, and approximately 1,000 to 1,500 feet inland on the west side.
- ***Bay Sediment Unit:*** The BSU consists of a semi-confining layer in areas where silts and clays of the BSU are of sufficient thickness. The BSU acts as a confining to semi-confining layer between the FWBZ and the underlying SWBZ. The BSU is not an effective aquitard in the southeastern portion of Alameda Point, where it is thin or not present at all.
- ***Second Water Bearing Zone:*** The SWBZ is confined to semi-confined groundwater in Merritt Sand (where present) and the upper unit of the San Antonio Formation. The SWBZ is divided into two hydrogeologic subintervals: The SWBZ-Upper (comprised of Merritt Sand) and the SWBZ-Lower (comprised primarily of the Upper San Antonio Formation). Where the BSU is thin or absent, hydrologic isolation between the FWBZ and the SWBZ has not been clearly established. Where the BSU is the predominant lithologic unit representing deposition within paleochannels in the central portion of Alameda Point, the FWBZ-SWBZ zones may not be clearly defined. The SWBZ is strongly tidally influenced in the central, southwest, and southeast portions of Alameda Point.
- ***Yerba Buena Mud Aquitard:*** The Yerba Buena Mud Aquitard is a regionally continuous layer that forms a well-known regional aquitard. At Alameda Point, the unit is approximately 35 feet thick and is encountered at depths between 80 and 120 feet bgs. The Yerba Buena Mud Aquitard is an effective confining layer between the relatively saline groundwater of the SWBZ and the underlying freshwater Alameda Formation Water- Bearing Zone.
- ***Alameda Formation Water-Bearing Zone:*** This is the deep water-bearing unit underlying the Yerba Buena Mud Aquitard in Alameda Formation sediments. The Alameda Formation Water-Bearing Zone is encountered at depths between 115 and 155 feet bgs.

For the purposes of the BGMP, in the areas where the BSU is present, the aquifers are referred to as the FWBZ and SWBZ. In areas where the BSU is absent or not laterally continuous, the unconfined aquifer is referred to as the Shallow Water-Bearing Zone (WBZ) (approximately equivalent to the FWBZ) and the Deep WBZ (approximately equivalent to the SWBZ); however, the Shallow WBZ can be subdivided into a shallow portion and a deeper portion dependent on interpretations of the local hydrogeology at each separate OU or IR site. For example, at OU-2B the feasibility study states that the unconfined/semi-confined WBZ extends to at least 70 feet bgs (SES-TECH, 2015). Therefore, the Shallow WBZ at OU-2B includes wells screened to a depth of 70 feet bgs.

Groundwater in most of the FWBZ and the Shallow WBZ in the western half of Alameda Point is slightly saline. Groundwater at Alameda Point generally flows from the middle of Alameda Point

SAP WORKSHEET #10 CONTINUED

outward toward San Francisco Bay, the Oakland Inner Harbor, and Seaplane Lagoon. Groundwater flow directions may vary throughout the year because of seasonal changes in precipitation and tidal changes (ITSI, 2009).

Problem Definition

Various compounds exceeding comparison criteria and remedial goals remain in groundwater at OU IR sites across Alameda Point.

Problem Statement

Previous investigations under CERCLA have documented releases of hazardous chemical to subsurface groundwater at OUs and IR sites across the former base. Previous treatability studies, and removal and remedial actions have addressed these sources of chemical release in soil and groundwater, and have significantly reduced the concentrations. The BGMP is designed to continue to monitor concentrations of the chemicals of concern at OUs and IR sites where remediation efforts have not been completed. The BGMP thus provides supplemental information for ongoing remedial action and monitored natural attenuation evaluations, and additional groundwater data for post-ROD.

SAP WORKSHEET #11 PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

This worksheet provides Steps 2 through 7 of the DQO process as detailed by the U.S. EPA (U.S. EPA, 2006). This process is used to determine the type, quantity, and quality of data necessary to support decision making regarding current site conditions and future site management decisions. The information inputs and analytical approach that will be used to achieve the objectives and the performance criteria will be used to ensure that the data used to make project decisions are of sufficient quality.

Step 2: Identify the Goals of the Program

The goals of BGMPs are to support groundwater and soil gas monitoring recommended/required by CERCLA remedial decisions, including Monitored Natural Attenuation (MNA) and LTM, at OUs and IR sites at Alameda Point. These goals will be achieved by performing groundwater and soil gas sampling, monitoring groundwater elevations at wells sampled as part of the BGMP, performing additional well installation and hydropunch installation, and performing well surveying, maintenance, and decommissioning as necessary. The BGMP will also be used to optimize the monitoring program.

Groundwater Sampling: A groundwater sampling program is in place to analyze compounds and geochemical parameters at or above their screening criteria.

These goals will be achieved by answering the following specific decision questions:

a. Do chemicals of concern (COCs) identified in the RODs persist above their remediation goals in groundwater?

b. Do groundwater MNA parameters support continuous degradation of COCs in groundwater and progress toward meeting site mediation goals?

c. Do the existing BGMP groundwater monitoring wells and chemical analytes provide adequate characterization of groundwater conditions to support site decisions?

Soil Gas Monitoring: Soil gas monitoring is conducted and is collected to provide data on the nature and extent of landfill-related contaminants.

a. Are contaminants present in soil gas at concentrations at or above levels defined in decision documents and monitoring plans?

b. Does the existing BGMP for soil gas monitoring well sampling provide adequate characterization to support CERCLA actions?

BGMP Optimization: in an effort to maximize the efficiency of the monitoring program at Alameda Point, each quarter the existing network of wells and analytical suites will be evaluated with respect to data collected over the previous 5 years, and evaluated for additions or subtractions of wells, analytical suites, and other monitoring activities.

a. Do the existing BGMP groundwater monitoring wells provide adequate characterization of groundwater conditions to support proposed or ongoing CERCLA actions?

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b. Are there analytes at or below their cleanup goal that can be removed from the analytical suites?

c. Has a sufficient amount of data been collected that monitoring frequency can be reduced?

Step 3: Identify Information Inputs

Information inputs will include results of analytical testing of groundwater and soil gas samples collected from OU and IR sites currently at Alameda Point and historical data from past years monitoring programs. Chemicals that may be found in groundwater and soil gas are identified in Step 5.

Step 4: Identify the Boundaries of the Study

The lateral boundaries of this project are covered by seven Alameda Point OUs (OU-1, OU-2A, OU-2B, OU-2C, OU-3, OU-4A, and OU-6) and 24 IR sites shown on Figure A-2. Zoomed-in views of each IR site depicting the wells associated with the site that may be part of the BGMP are shown on Figures A-5 through A-25. Soil gas monitoring locations are shown on Figure A-26.

The vertical boundaries of the Alameda Point study area are the bottoms of the FWBZ and the SWBZ, depending on the geographic location and the depth of groundwater impact. The vertical boundary for each well depends on the well construction and depth to water, which ranges from approximately 2 to 100 feet bgs at Alameda Point. The vertical boundaries for the soil gas wells is approximately 1 to 15 feet bgs based on previous monitoring at IR Sites 1, 2, and 32.

The temporal boundaries of the BGMP will vary with each site, and may not be fixed until approval of a site's remedial design monitoring plan, or as determined by the BCT using guidelines described in this BGMP.

Step 5: Develop the Analytical Approach

Groundwater samples collected for chemical analysis will provide the basis for monitoring dissolved concentrations of compounds in groundwater, provide supplemental information for ongoing remedial action and monitored natural attenuation evaluations, and to provide additional groundwater data post-ROD and pre-remedial action at various groundwater monitoring sites. These analytes may include:

- TPH-e (diesel, jet fuel, and motor oil) by U.S. EPA Method 8015B
- TPH-p (gasoline) by U.S. EPA Method 8015B
- VOCs by U.S. EPA Method 8260B
- Polynuclear Aromatic Hydrocarbons (PAHs) by U.S. EPA Method 8310
- 1,4-Dioxane by U.S. EPA Method 8270 SIM
- SVOCs by U.S. EPA Method 8270C
- Pesticides (by U.S. EPA Method 8081
- PCBs by U.S. EPA Method 8082
- Dissolved Metals by U.S. EPA Methods 6010B/6020A/7470A
- Hexavalent Chromium (by U.S. EPA Method 7196A
- Cyanide by U.S. EPA Method 335.2

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- Dissolved Gases (methane, ethane, ethene) by SOP RSK 175
- Anions (nitrate, nitrite, chloride, sulfate) by U.S. EPA Method 300.0
- Alkalinity by U.S. EPA Method 310.2
- Sulfide by U.S. EPA Method 376.2
- Ferrous Iron by Hach Test Kit
- TDS by U.S. EPA Method 150.1
- BOD by U.S. EPA Method 405.1
- COD by U.S. EPA Method 410.4
- TOC by U.S. EPA Method 415.1
- DOC by SM 5310C
- Census DNA by qPCR
- Gross Alpha, Gross Beta by U.S. EPA Method 9310/900.0
- Radium-226 by U.S. EPA Method 9315/903.0
- Tritium by U.S. EPA Method 906.0
- Total Radium by U.S. EPA Method 904.0
- Strontium-90 by U.S. EPA Method 905.0
- Gamma Emitting Radionuclides (Cs-137 and Co-60) by U.S. EPA Method 901.1
- Uranium Isotopes by U.S. EPA Method 908 Modified
- Water Quality parameters:
  - ORP using a field meter
  - pH using a field meter
  - DO using a field meter and Hach Test Kit
  - Temperature using a field meter
  - Specific Conductance using a field meter

Soil Gas:

- Fixed Gases by U.S. EPA Method TO-3
- VOCs by U.S. EPA Method TO-15

Based on the analytical approach and the analyses listed above, the following decision rules that address each decision question in Step 2 are proposed:

Groundwater Sampling:

1. **If** reported concentrations of COC for the given IR/OU site are below their remedial goals identified in the RODs, **then** a recommendation may be made that the OU/IR site has met its cleanup goal and no further sampling will be required as part of the BGMP. If reported concentrations of COC for the given IR/OU site are above the comparison criteria, **then** monitoring at the site will be continued.

2. **If** MNA parameters analyzed support continuous degradation of COCs in groundwater and progress toward meeting site remediation goals, **then** the data will be used for optimization of the BGMP including reducing sampling frequency.

3. **If** groundwater monitoring well layout does not adequately characterize groundwater conditions to support CERCLA actions, **then** additional monitoring wells may be added to the BGMP.

SAP WORKSHEET #11 CONTINUED

Soil Gas Monitoring:

1. **If** contaminants are not detected for the given IR site, or the measured concentration of contaminant is below the comparison criteria, **then** a decision may be made that the IR site is adequately characterized with respect to soil gas and reduced monitoring frequency will be proposed as part of the BGMP. Otherwise, **if** contaminants are detected in the IR site samples tested at concentrations greater than or equal to the comparison criteria, **then the** data may be compiled for use by others to design remedial efforts and/or to evaluate potential risks to human health and the environment.

2. **If** the soil gas monitoring network does **no**t adequately delineate the plumes, **then** additional soil gas probes may be proposed.

BGMP Optimization:

a. **If** the existing BGMP groundwater monitoring wells provide adequate characterization of groundwater conditions to support proposed or ongoing CERCLA actions, **then** the well network will be evaluated to identify wells where sampling is no longer necessary.

b. **If** there analytes at or below their cleanup goal that can be removed from the analytical suites, **then** the analytes will be removed from the monitoring program at that specific OU and IR site.

c. **If** a sufficient amount of data been collected that monitoring frequency can be reduced, **then** the monitoring frequency will be reduced for that specific OU and IR site.

Step 6: Specify Performance or Acceptance Criteria

Sampling objectives to support site decisions for groundwater and soil gas limit the use of statistical methods in selecting sampling locations for this BGMP. Sampling locations are based on professional judgment and are designed to provide adequate characterization of the OUs and IR sites. Tolerable limits on decision errors cannot be precisely defined.

To limit uncertainty in the obtained environmental data, criteria have been developed for the precision, accuracy, representativeness, completeness, and comparability parameters and limit of detection (LOD) for the chemicals of concern as presented in Worksheet #37. Measurement errors will be controlled by using appropriate sampling and analytical methods; laboratory errors will be controlled by adhering to established standard operating procedures (SOPs) and having third-party data validation to verify laboratory processes. The field crews will review the SAP before sample collection to apply consistent sampling methods and limit sample collection errors. The subcontracted analytical laboratory will have a copy of the SAP and will follow DoD QSM guidance to limit measurement errors.

Step 7: Develop the Plan for Obtaining Data

Groundwater Monitoring: Groundwater samples will be collected from existing permanent groundwater monitoring wells and temporary hydropunch locations, and may be analyzed for compounds identified in the list of analytical methods in Step #5 above. Data collection and analysis are described in Worksheets #14 and #17 and will satisfy the DQOs specified in the preceding six steps. Analytical data will be validated by a third-party data validator.

Soil Gas Monitoring: Soil gas samples will be collected from existing permanent soil gas wells using standardized sampling techniques. The samples will be analyzed for fixed gasses

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(including carbon dioxide [CO2], oxygen [O2], nitrogen [N2], and carbon monoxide [CO]) and for VOCs, including methane, ethane, and ethene, on an annual basis.

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SAP WORKSHEET #12 MEASUREMENT PERFORMANCE CRITERIA TABLE

Table 12-1. Measurement Performance Criteria – Soil Gas

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Field Duplicate	Fixed Gases; Methane, Ethane, Ethene	10%	Precision and Accuracy	RPD ≤50% when detected concentrations >5x QL; difference ≤QL if concentrations < 5x QL	S&A

Notes:
(see Table 12-2 below)

Table 12-2. Measurement Performance Criteria – Groundwater

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Field Duplicate	Analytical Suite 1	10%	Precision and Accuracy	RPD ≤30% when detected concentrations >5x QL; difference ≤QL if concentrations < 5x QL	S&A
Equipment Blanks	Analytical Suite 1	One daily per sampling team for wells sampled without dedicated equipment.	Accuracy/Bias, Contamination	No target analytes > QL	S
Source Blanks	Analytical Suite 1	One per sampling event	Accuracy/Bias, Contamination	No target analytes > QL	S
Trip Blanks	VOCs	One per cooler containing VOC samples	Accuracy/Bias, Contamination	No target analytes > QL	S
Temperature Blank	Not Applicable	One per cooler	Accuracy/ Bias	≤6ºC when received at laboratory	S

Notes:
RPD = Relative Percent Difference
QL = Quantitation Limit
Analytical Suite 1 includes TPH, VOCs, 1,4-dioxane, SVOCs, PAHs, pesticides, PCBs, metals, cyanide, and radionuclides.

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SAP WORKSHEET #13 SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

Secondary Data	Data Source (originating organization, report title, and date)	Data Generator(s) (originating organization, data types, data generation/collection dates)	How Data Will Be Used	Limitations on Data Use
Sampling and Analysis Plan	AMEC, *Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan),* Alameda Point, Alameda, California. August 2010.	AMEC	Data will be used to evaluate assumptions regarding site conditions and the nature and extent of contamination as well as establish guidelines for the optimization of BGMP sampling/monitoring	SAP amended by Addendum 1 in December 2010
Sampling and Analysis Plan – Addendum 1	SES-TECH, *Final Addendum 1 to the Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan),* Alameda Point, Alameda, California. December 2010.	SES-TECH	Data will be used to update assumptions regarding site conditions and the nature and extent of contamination and additional tasks under the BGMP	Addendum 1 amended by Addendum 2 in November 2011
Sampling and Analysis Plan – Addendum 2	SES-TECH, *Final Addendum 2 to the Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan),* Alameda Point, Alameda, California. November 2011.	SES-TECH	Data will be used to update assumptions regarding site conditions and the nature and extent of contamination and additional tasks under the BGMP	Addendum 2 amended by Addendum 3 in November 2012
Sampling and Analysis Plan – Addendum 3	SES-TECH, *Final Addendum 3 to the Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan),* Alameda Point, Alameda, California. November 2012.	SES-TECH	Data will be used to update assumptions regarding site conditions and the nature and extent of contamination and additional tasks under the BGMP	Addendum 3 amended by Addendum 4 in January 2014

SAP WORKSHEET #13 CONTINUED

Secondary Data	Data Source (originating organization, report title, and date)	Data Generator(s) (originating organization, data types, data generation/collection dates)	How Data Will Be Used	Limitations on Data Use
Sampling and Analysis Plan – Addendum 4	SES-TECH, *Final Addendum 4 to the Final Sampling and Analysis Plan (Field Sampling and Quality Assurance Project Plan),* Alameda Point, Alameda, California. January 2014.	SES-TECH	Data will be used to update assumptions regarding site conditions and the nature and extent of contamination and additional tasks under the BGMP	No limitations on data
2004 Radionuclide Evaluation	Shaw, Evaluation of Radiological Groundwater Analyses	Shaw	Established parameters will be used for evaluating radionuclides in groundwater	Concentrations may vary between FWBZ and SWBZ
2004 Tidal Study	Shaw, Tidal Study for IR Sites 1 and 2	Shaw	Data will be used to determine appropriate time for measuring groundwater levels in wells subject to tidal influences	BGMP wells have been added subsequent to this study

SAP WORKSHEET #14 SUMMARY OF PROJECT TASKS

The MMEC Group has been contracted by the Navy to perform BGMP to monitor impacted subsurface water and soil gas at 24 IR Sites and seven OUs at Alameda Point. The monitoring program consists of groundwater sampling, soil gas sampling, well installation, hydropunch sampling, well maintenance, surveying, and well decommissioning at various OU and IR sites across Alameda Point (Figure A-3).

14.1 Permitting and Notification

Permits for the decommissioning of the identified wells will be acquired through the Alameda County Environmental Health Department. All well decommissioning activities will be performed under the guidance and supervision of a California Professional Geologist (PG), Certified Engineering Geologist (CEG), or Professional Engineer (PE) in accordance with California Department of Water Resources Bulletin 74-90, and any applicable local regulations.

Before the beginning of field work, the MMEC Group will inform the Navy of the nature and schedule of the planned field work. The MMEC Group will coordinate site access through the BRAC Caretaker or Activity POC, and will notify the ROICC of the upcoming schedule for field work.

Coordination of sampling activities and required QC measures will take place between the MMEC Group and the Navy RPM regarding contractors performing work at individual IR Sites prior to initiating the field sampling activities. Daily discussions between the field team leader, the quality officer, and the field team staff will take place prior to any sampling activities. These meetings will entail a discussion of assignments for the field team including which wells and what analytical groups will be sampled for the day. Additionally, end of day meetings will take place to catalogue that all assignments were implemented correctly.

Underground Service Alert (USA) will be notified of the intended activities at least 3 business days prior to the initiation of any invasive work. Alameda Utilities will also be notified prior to intrusion activities.

14.2 Mobilization

Mobilization for the field work will include designating a decontamination area and conducting a preparatory inspection. The preparatory phase inspection may be held prior to mobilization to discuss project scope, health and safety requirements, sampling procedures, status of submittals and procurements, and quality control protocols. A perimeter will be installed and used as a support zone, as needed. The IDW storage area will be designated through coordination with the RPM, POC, and ROICC.

14.3 Groundwater Monitoring

Groundwater from monitoring wells will be sampled for offsite analysis by a Department of Defense and California-approved laboratory, as described below. Monitoring wells will be purged and sampled in accordance with U.S. EPA groundwater sampling operating procedure (U.S. EPA, 2010a) or using a passive sampling method such as the Snap Sampler. The low-flow (*low-stress*) groundwater sampling purge rate of between 100 to 500 milliliters per minute (mL/min) will be employed, with a preference for the lower end of this range during the initial stages of purging. The overall goal of low-flow purging is to minimize water table drawdown, and appropriate procedures will be followed during low-flow purging operations. A submersible pump

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(bladder pump) with a flow controller and disposable tubing and bladders will be used for sample collection. The following procedures will be used at each well prior to sampling.

1. Assemble well location maps, construction information, keys, and sampling equipment and transport them to the site.
2. Water level monitoring and sampling must begin at the monitoring well with the least contamination, generally up-gradient or farthest from the site or suspected source. Then proceed systematically to the monitoring wells with the most contaminated groundwater.
3. Check and record the condition of the monitoring well for damage or evidence of tampering. Lay out polyethylene sheeting around the well to minimize the likelihood of contamination of sampling equipment from the soil and exposure of soil to liquids dripping from the sampling equipment.
4. Unlock the well head. Record location, time, date, and appropriate information in a field logbook or on the groundwater sampling log.
5. Remove the inner casing cap.
6. Monitor the headspace of the monitoring well at the rim of the casing for VOCs with a photoionization detector (PID) and record in the logbook or on the sampling log. If the existing monitoring well currently has or has a history of positive headspace readings, then the sampling must be conducted in accordance with the Site Safety and Health Plan.
7. Measure the depth to water (water level must be measured to the nearest 0.01 foot) relative to a reference measuring point on the well casing with an electronic water level indicator or other appropriate measuring device, and record in logbook or on the sampling log. If no reference point is found, measure relative to the top of the inner casing, then mark that reference point and note that location in the field logbook. Record information on depth to groundwater in the field logbook or sampling log. Measure the depth to water a second time to confirm the initial measurement; measurements should agree within 0.01 foot, or re-measure.

Check the available well information or field check for the total depth of the monitoring well

14.3.1 Low-Flow Purging

Existing monitoring wells that will be identified in Worksheet #18 on a quarterly basis will be purged prior to groundwater sampling. The purging and groundwater sampling will be conducted in general accordance with the U.S. EPA low-flow purging and sampling methodology (U.S. EPA, 2010b). All sampling equipment will be thoroughly decontaminated prior to initiating any site work. All reusable sampling equipment will be decontaminated prior to use at each boring location to minimize the potential for contaminant migration or cross-contamination. Table 14-1 presents details on the existing monitoring well network that will be used in the BGMP. Figure A-3 shows the location of the wells.

Each well will be micro-purged (100 to 500 mL/min) using a bladder pump or equivalent. The pump should provide consistent results and minimal disturbance of the sample across low-flow rates.

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The following procedures will be followed when sampling a well:

1. Confirm the well identification at each monitoring well. Preferentially collect samples from wells with the lowest expected contaminant concentrations to the highest expected concentrations to minimize the potential for cross-contamination.
2. Put on a new, clean, and chemical-resistant pair of disposable gloves. Calibrate field instruments in accordance with the manufacturer's directions. Record all calibration documentation in the field logbook or on the "Groundwater Monitoring Form."
3. Measure the depth to water at each monitoring well using an electronic water level indicator probe. Record the water level measurement to the nearest 0.01 foot on the "Groundwater Monitoring Form." Decontaminate the water level indicator before each monitoring well measurement according to the decontamination procedure.
4. Carefully lower the tubing or the pump into the well with as little disturbance to the groundwater as possible. If the water level is above the top of the screen, place the intake to the pump at the middle of the screen interval. Install the pump slowly to the middle or slightly above the middle of the screened interval, thus minimizing mixing of stagnant casing water with formation water within the screened interval, and re-suspension of settled solids from the bottom of the well. If the water level is below the top of the screen, the pump intake will be placed in the middle of the water column.
5. Purge the well at a flow rate of 100 to 500 mL/min. Monitor water quality parameters (turbidity, pH, temperature, conductivity, ORP, and DO) every 3 to 5 minutes during purging, using in-line monitoring equipment to increase the reading stability. Record the water quality parameters on the groundwater sampling log form. Stabilization is achieved if three consecutive readings are within ± 0.1 pH units, ± 3 percent of the reading for temperature, ± 3 percent of the reading for conductivity, ±10 millivolts (mV) for ORP, ± 10 percent for DO (if DO reading is > 0.5 milligrams per liter [mg/L]), and ±10 percent for turbidity (if turbidity reading is > 5 Nephelometric turbidity units [NTU]).
6. When the water quality parameters are stable for three consecutive readings, collect samples for chemical analysis. If the water quality parameters have not stabilized, continue purging until stabilization occurs or until three calculated well volumes have been purged.
7. Samples will be collected at the same pump flow rate used during purging. Collect samples for VOC analysis first. Fill the containers so that no headspace exists. Collection of the total petroleum hydrocarbons-purgeable (TPH-p) sample will be next, followed by the remaining analyses. Collect field QC samples (e.g., field duplicates) as required.
8. Label samples properly and place the sample container into a ziplock bag. Transfer the samples to cold storage immediately after collection. Handle and transport the samples to the laboratory according to the procedure described in Worksheet #27.

14.3.1.1 Purging and Sampling Low-Yielding Groundwater Monitoring Wells

This purging and sampling method will be used for groundwater monitoring wells that do not produce sufficient water to meet the drawdown criteria of the low-flow method. This method will

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be utilized only after is has been demonstrated that drawdown criteria cannot be achieved using low-flow methods. The following steps will be taken to implement this sampling method:

1. Before well purging, the water level in the well will be measured with a water level meter. Water levels will continue to be measured during well purging activities. The well will be purged until the water level is even with the top of the pump or until one well casing column has been purged, whichever comes first. The pump will be placed in the approximate middle of the water column within the well screen interval (but no closer than 2 feet from the bottom of the well) to avoid mobilization and entrainment of settled solids from the bottom of the well. If one well casing volume is purged, groundwater samples will be then be collected. If the well is purged until the water level is even with the top of the pump, and a casing volume has not been purged, the well will be secured and allowed to sit until a sufficient column of groundwater has entered the well to enable to collection of groundwater samples (re-purging should be performed if a well is inactive for more than 24 hours).
2. The water level will be monitored until the well has recharged to approximately 80 percent of the pre-purge water level.
3. The flow-through cell used for water quality parameter measurements will be disconnected before any samples are collected.
4. Groundwater samples will then be collected at the purging rates specified below in this worksheet.
5. The well will be secured and the sampling team will demobilize.

14.3.1.2 Purging and Sampling Groundwater Monitoring Wells Containing Residual Non-aqueous Phase Liquid

This purging and sampling method will be used for sampling groundwater monitoring wells where residual non-aqueous phase liquid (NAPL) has the potential to contaminate sampling equipment. The following steps will be taken to implement this sampling method:

1. A sample of NAPL will be collected without purging the well. A light non-aqueous phase liquid (LNAPL) sample will be collected using a bottom valve bailer lowered to a depth less than the LNAPL/water interface. A dense non-aqueous phase liquid (DNAPL) sample will be collected using a bailer lowered to the bottom of the well.
2. No water quality measurements will be collected, as residual NAPL has the potential contaminate/damage the flow-through cell.
3. It may not be meaningful to conduct laboratory analysis of a groundwater sample collected from a well containing NAPL. The NAVFAC SW RPM and MMEC Group PM will be consulted if NAPL is detected and before groundwater samples are analyzed.

14.3.1.3 Groundwater Sample Collection

Groundwater samples will be collected in containers provided by the laboratory that are of sufficient volume and contain preservatives, as appropriate, for the analytical methods. To prevent cross-contamination, samples will be transferred directly into the sample containers from the pump discharge tubing, not through flow meters or flow-through cells. Sample containers will be filled as follows:

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1. Volatile organic analysis (VOA) vials for VOCs and TPH-p analysis will be filled first. The VOA vials will be filled so that no headspace (air bubbles) is present, and will be field-checked for headspace. If air bubbles are observed within a VOA vial, the sample will be discarded and a new sample will be collected in a new VOA vial. The purge rate for these analyses will be 100 mL/min or less, to minimize loss of volatile constituents. Groundwater samples will be collected from the well discharge tubing either from disposable T- or Y-fittings places near the inlet to the flow-through cell, or after disconnecting the flow-through cell. The VOA vials from each well will be immediately stored together in re-sealable plastic bags to prevent cross-contamination with other groundwater samples.
2. The samples for remaining analysis will then be collected at a purge rate of 500 mL/min or less.
3. Samples collected for dissolved metals will be field-filtered or laboratory-filtered before analysis. If samples are field-filtered, they will be filtered using a new 0.45-micron disposable plastic filter placed on the end of the discharge tubing, and collected at a sampling rate of 500 mL/min or less. To minimize the potential for cross-contamination, one filter will be used at each well and then discarded.

After sample collection is complete, the sample containers will be immediately labeled and placed in the cooler, to be maintained at a temperature of 6 degrees Celsius (°C) or below.

14.3.2 SNAP Sampler

The following procedures describe the deployment and sampling for the Snap Sampler® passive groundwater sampling method. These procedures describe steps for dedicated and non-dedicated systems.

14.3.2.1 Deployment Activities

Selection of the deployment depth within the screen interval is dependent on site-specific conditions. Normally, deployment depth is targeted at the center of the well screen.

If depth-specific monitoring is desired, multiple samplers may be deployed at intervals appropriate for the sampling objective.

If multiple sample zones within a well, zone isolation using a packer or baffle device to limit in-well mixing can be used (Britt, 2006; Britt and Calabria, 2008). These can be attached to the Snap Sampler® trigger line or deployed separately. Installation of an upper baffle designed to isolate the unscreened well casing or well headspace may be desired. The upper baffle will limit mixing of “stagnant” casing water with screen-interval water, and/or gas exchange with the headspace air.

1. Remove the Snap Sampler bottle(s) from its packaging.
2. Turn the translucent (PFA) vial cap on each end of the bottle slightly to release the o-ring.
3. Insert the bottle into the upper end of the sampler.
4. Place the sampler connector onto each end of the sampler; turn clockwise to align the set pins/screw; then gently tighten the set screw with the Snap Driver Tool.

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5. Pivot the vial cap (Snap Cap) into its seat with the Snap driver. Push up the retainer pin through the lower hole in the vial cap. Repeat for all Snap Caps. If an O-ring should dislodge from its seat during setting, remove the sample bottle and carefully replace it in the o-ring groove; repeat setting procedure.
6. For the manual trigger, feed ball-fitting end of trigger cable through lower release pin groove; click tube fitting into connector.
7. Press in the ball fitting to attach to lower release pin.
8. For the electric or pneumatic trigger system, attach the wireline from the plunger.
9. Deploy to selected depth with trigger cable/tubing and attach to well head docking station.
10. Additional Snap Samplers® can be deployed with separate trigger tubing cables or in series with a single trigger. If separate triggers are used, the ID tags should be marked at the surface for later reference.
11. The recommended minimum deployment period is two weeks. There may be hydrogeologic conditions where a shorter deployment is possible, but two weeks would generally assure a return of the well to its steady-state condition (Vroblesky, 2001a, 2001b). Parker et al. (2009) found that a 72-hour submergence time was sufficient for most analytes tested, but this does not account for well disturbance or other well-specific factors. The user may determine that shorter deployment times than 2 weeks are appropriate for a specific application.
12. The Snap Sampler® can be deployed for extended periods. No upper bound for sampler deployment has been found, rather, conditions at individual wells seem to control the applicability of deployments lasting a year or longer.

14.3.2.2 Sample Collection Activities

When the deployment interval is completed, the sampler should be triggered at the well head without disturbing the sampler position. For the manual trigger, the cable end should be pulled with sufficient force to move the cable up the tubing. Depending on the length of the cable, closure of the samplers usually can be felt through the trigger line when the samplers trip. If more than one triggering line is present, closure should proceed from the deepest to the shallowest sampler position to limit capture of sediment potentially re-suspended by closure of the first sampler.

After the sampler is triggered and retrieved, the upper connector should be removed by loosening the retainer screw and turning the connector. The bottom connector piece does not need to be disassembled to remove the bottles.

While the bottles should not leak with reasonable handling, they should not be agitated (to check for headspace, for example) until after the screw caps are tightened. Under most circumstances there will be no air in the vials at retrieval. However, some field conditions—including deep groundwater, natural effervescence, or other causes—may cause some small air bubbles to be present in the bottle or on the spring when retrieved. This is not a concern if the air was entrained while deployed. Air adhering to the vial during deployment would be in equilibrium with the sample water upon sampler closure. Therefore it is not “headspace air” into

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which sample VOCs could volatilize. Deployment air could be attached to the spring or cap, and should not be larger than 1-2 millimeters (mm) upon retrieval. Pankow (1986) showed that small headspace air from these or other causes do not substantially impact results for most common volatiles. If air bubbles are larger than 5 mm before placing the screw cap, or water is clearly leaking from the vial, the sample may not have sealed properly and the sample container will be discarded.

There are no special laboratory preparation procedures for Snap Sample bottles. The bottles can be analyzed using common 40-mL autosamplers. The spring inside the VOAs is polymer-coated and will deflect away from the autosampler extraction needle during insertion.

14.3.2.3 Decontamination Procedures

The electronic water level indicator probe/steel tape, the water quality field parameter sensors and any *non-dedicated* Snap Sampler® groundwater sampling equipment should be decontaminated by the following procedures:

1. The water level meter will be hand washed with phosphate-free detergent and a scrubber, then thoroughly rinsed with distilled water.
2. Water quality field parameter sensors with distilled water between sampling locations will be utilized. No other decontamination procedures are necessary or recommended for these probes since they are sensitive. After the sampling event, the sensors must be cleaned and maintained per the manufacturer's requirements.
3. For *non-dedicated* applications, the Snap Sampler® and trigger tubing must be pressure-sprayed or bristle-brush scrubbed with soapy water, tap water, and distilled water prior to use in a different well. Depending on the condition of the Snap Sampler®, the release pin mechanism may need to be disassembled to effectively clean the pins and grooves. Disassembly can be accomplished by removing the lever screw.

14.4 Analytical Tasks

The analytical parameters and methods for the BGMP were selected on the basis of available historical information regarding previous site investigations. Not all analytical methods listed below will be used at every IR site.

Groundwater samples collected from groundwater monitoring wells at the various OU and IR sites will be analyzed in a fixed laboratory or in the field using a field meter or test kit for the following:

- TPH-e (diesel, jet fuel, and motor oil) by U.S. EPA Method 8015B
- TPH-p (gasoline) by U.S. EPA Method 8015B
- VOCs by U.S. EPA Method 8260B
- Polynuclear Aromatic Hydrocarbons (PAHs) by U.S. EPA Method 8310
- 1,4-Dioxane by U.S. EPA Method 8270 SIM
- SVOCs by U.S. EPA Method 8270C
- Pesticides (by U.S. EPA Method 8081
- PCBs by U.S. EPA Method 8082
- Dissolved Metals by U.S. EPA Methods 6010B/6020A/7470A
- Hexavalent Chromium by U.S. EPA Method 7196A

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- Cyanide by U.S. EPA Method 335.2
- Dissolved Gases (methane, ethane, ethene) by SOP RSK 175
- Anions (nitrate, nitrite, chloride, sulfate) by U.S. EPA Method 300.0
- Alkalinity by U.S. EPA Method 310.2
- Sulfide by U.S. EPA Method 376.2
- Ferrous Iron by Hach Test Kit
- TDS by U.S. EPA Method 150.1
- BOD by U.S. EPA Method 405.1
- COD by U.S. EPA Method 410.4
- TOC by U.S. EPA Method 415.1
- DOC by SM 5310C
- Census DNA by qPCR
- Gross Alpha, Gross Beta by U.S. EPA Method 9310/900.0
- Radium-226 by U.S. EPA Method 9315/903.0
- Tritium by U.S. EPA Method 906.0
- Total Radium by U.S. EPA Method 904.0
- Strontium-90 by U.S. EPA Method 905.0
- Gamma Emitting Radionuclides (Cs-137 and Co-60) by U.S. EPA Method 901.1
- Uranium Isotopes by U.S. EPA Method 908 Modified
- Water Quality parameters:
  - ORP using a field meter
  - pH using a field meter
  - DO using a field meter and Hach Test Kit
  - Temperature using a field meter
  - Turbidity using a field meter
  - Specific Conductance using a field meter

Refer to Worksheet #18 for the analyses to be conducted at each well, as not all analyses will be performed in every well. No radiological samples are currently planned for the BGMP; however, if radiological sampling in the future is required then RASO will be contacted.

14.5 Water Level Measurements

Groundwater levels will be measured at the monitoring wells to be sampled during each quarterly BGMP event immediately prior to purging and sampling activities.

At each well, the well cap will be removed and the wellhead will be checked for organic vapors using a PID. A permanent reference mark has been located or scribed onto the top of the casing to provide a consistent reference point from which all levels are measured. Depth to water (DTW) measurements will be taken to an accuracy of 0.01 foot using a water-level indicator. The water-level indicator will be decontaminated as described below. The measurements will be checked by slowly raising and lowering the tape and watching the instrument response. The measurement will be recorded in the field logbook.

14.6 Soil Gas Sampling

The soil gas wells identified for sampling will be purged prior to soil gas sampling. All sampling equipment will be thoroughly decontaminated prior to initiating any site work. All reusable sampling equipment will be decontaminated prior to use at each location to minimize the

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potential for contaminant migration or cross-contamination. Figure A-26 shows the location of the soil gas wells.

Each sampling and dedicated tubing assembly will be purged of 1- to 7-system volumes of "dead space volume" and leak checked prior to collection of the soil gas sample using a rotometer and Summa canister.

The following procedures will be followed when sampling a soil gas well:

1. Confirm the well identification at each monitoring well.
2. Put on a new, clean, and chemical-resistant pair of disposable gloves. Calibrate field instruments in accordance with the manufacturer's directions. Record all calibration documentation in the field logbook or on the "Soil Gas Monitoring Form."
3. Assemble the system sampling assembly including air pump, flow meter, in-line rotometer, 3-way valve, Teflon ® tubing, and Summa canister.
4. Calculate the purge volume and perform a leak test using an inert tracer compound such as helium at a flow rate of no greater than 200 mL/min.
5. If the leak detection testing indicates that there is no leak in the sample assembly, then perform the soil gas sampling by opening the 3-way valve to the Summa canister and collect the soil gas sample, making sure to record the canister vacuum and time. The sample collection flow rate should not exceed 200 mL/min.
6. Sample collection should terminate while a small vacuum still remains in the canister.
7. Close the Summa canister and label with a unique identifier, and record the final canister vacuum reading and time.
8. Collect field QC samples (e.g., field duplicates) as required.
9. Keep the Summa canister out of the sun and do not chill. Label samples properly and place the sample container out of the sun. Handle and transport the samples to the laboratory according to the procedure described in Worksheet #27.

Soil gas samples will be analyzed in a fixed laboratory for the following:

- Fixed Gases by U.S. EPA Method TO-03
- VOCs by U.S. EPA Method TO-15

14.7 Monitoring Well Decommissioning

Monitoring wells identified by the Navy for destruction will be decommissioned in accordance with California Department of Water Resources Bulletin 74-90, and any applicable local regulations. Well materials including, but not limited to, protective surface casings, bollards, traffic boxes, PVC or other riser pipe and/or screen, filter sand, bentonite seal material, annular seal, concrete, and/or cement fill material will be removed and disposed of in accordance with the MMEC Group current waste management plan. The decommissioning of the wells will be performed under the guidance of a California-registered Professional Geologist, Certified Engineering Geologist, or Professional Civil Engineer, by a drilling contractor with a valid State of California C-57 License.

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14.8 Decontamination

All sampling equipment will be thoroughly decontaminated prior to initiating any site work. All reusable sampling equipment will be decontaminated prior to use at each sampling location to minimize the potential for contaminant migration or cross-contamination. Equipment decontamination areas will be located within or adjacent to the field work area, as designated by supervising field personnel. Equipment decontamination will be conducted in accordance with the following procedure:

1. Potable water and nonphosphate detergent (i.e., Liquinox™) wash (using brushes or a steam cleaner, as appropriate)
2. Potable water rinse
3. Distilled or deionized water rinse
4. Equipment air dried and stored until used

The effectiveness of the decontamination procedures will be tested by collecting one equipment rinsate per day of use following the end-of-the-day equipment decontamination. The laboratory-supplied, analyte-free water will be poured over and around the decontaminated sampler and collected for analysis at the laboratory. Decontamination liquids will be managed along with other IDW as described below.

14.9 Well Installation

Well installation may be needed at Alameda Point as part of this BGMP in order to expand an existing well network or evaluate IR site conditions based on data collected under separate task orders and documented in other reports and planning documents. Prior to any ground disturbance, the site will be marked with white paint (or other appropriate marking technique) and USA and Alameda Utilities will be notified at least 48 hours in advance of the planned disturbance. Additionally, the MMEC Group may decide to employ a private utility locating service to clear the proposed disturbance area of underground utilities. Well materials including, but not limited to, PVC or other riser pipe and/or screen, filter sand, bentonite seal material, annular seal, concrete, and/or cement fill material will be installed in accordance with the permitting agency's guidelines, as well as in accordance with local, state, and federal regulations. The installation of the wells will be performed under the guidance of a California-registered Professional Geologist, Certified Engineering Geologist, or Professional Civil Engineer, by a drilling contractor with a valid State of California C-57 License.

14.10 Hydropunch Sampling

Hydropunch sampling may be needed in order to evaluate groundwater conditions at IR sites in areas where a groundwater monitoring well may not exist. The locations of hydropunch samples will be determined based on data collected under separate task orders and documented in other planning documents or thru evaluation of groundwater data collected as part of the BGMP at the direction of the Navy. Hydropunch sampling will provide a means to evaluate groundwater conditions at IR sites without having to install a permanent monitoring well. Grab groundwater samples will be collected from borings advanced by DPT using a HydroPunch sampling tool or equivalent. Grab groundwater samples may be collected from any water bearing zone at Alameda Point if needed. Once the targeted sampling depth is reached, the HydroPunch tool will be threaded onto a steel drive pipe and pushed several feet into the

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saturated formation. The drive pipe will be pulled upward to open the inlet of the sampler, allowing groundwater to flow into the screened sample chamber. Samples will be collected from the sample chamber using a stainless steel or disposable bottom-emptying bailer, or peristaltic pump. Water quality readings (dissolved oxygen, turbidity, salinity, pH, etc.) will be taken before the sample is collected. Grab groundwater samples will be collected in the appropriate sample container based on the analytical method requirements.

14.11 Well Maintenance Activities

Well maintenance activities, including repair or replacement of bolts, locks, J-plugs, well boxes (or components thereof), broken casings, and well monuments/pads, may be performed as part of the BGMP. The maintenance activities will be performed on wells that have been identified as requiring maintenance or repair through the BGMP. Repairs such as bolt, lock, or J-plug replacement may take place at the time of the observed deficiency. Repairs such as replacement of well boxes, well monuments/pads, or casings will require proper notification and possible mobilization of contractors to perform the maintenance/repair work. All work will be performed in accordance with the California Department of Water Resources Bulletin 74-90 and with applicable local regulations, and will be properly permitted.

14.12 Surveying

Newly installed wells and wells that have been repaired such that their elevations may have been altered will be surveyed. Surveying activities will be conducted by a California-licensed land surveyor. The northings, eastings, and elevations will be collected with a minimum accuracy of ± 0.01 foot. Elevations will be surveyed relative to mean sea level (msl).

14.13 Investigation-derived Waste Management

Waste management will follow the Waste Management Plan provided as Appendix B to the Work Plan. IDW generated during the field activities will include purged groundwater, decontamination fluids, personal protective equipment (PPE), and other disposable sampling materials. IDW generated during well installation and/or decommissioning may include: bentonite, grout, cement, concrete, PVC, decontamination fluids, and PPE. All IDW will be managed and disposed of in accordance with U.S. EPA and California Department of Transportation (DOT) guidelines in the Waste Management Plan provided as Attachment B to the Work Plan, and as summarized below.

Pending characterization, IDW will be stored onsite in a secure and controlled area. IDW will include the wastewater from decontamination procedures, which will be containerized in DOT-approved 55-gallon steel drums and appropriately labeled until waste characterization is complete. IDW generated in IR Sites 1, 2, or 32 will remain within the controlled area until approved for removal and disposal, at which time the waste will be physically scanned out of the controlled area by a radiation technician.

A small amount of nonhazardous PPE and sampling equipment IDW will be generated during the field work. Used PPE may consist of protective coveralls, nitrile gloves, and other disposable gear associated with field activities. Sampling equipment may include such items as sampling tools, cleaning/decontamination equipment, and used paper towels. Used PPE will be stored onsite, double-bagged, and disposed of along with other nonhazardous solid waste pending the analytical results of the field samples. Disposal of IDW will be performed within 90 days of waste

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generation. The removal of IDW to an offsite disposal facility will be coordinated with the NAVFAC SW RPM and the Alameda Point ROICC.

14.14 Field Documentation

Field measurements will be made by qualified Field Team Leaders, Field Geologists, Engineers, Environmental Scientists, and/or Technicians. All field data will be recorded in ink on the forms listed below. Review of field data and records will be performed by the Field Team Leader or designee. Instrument selections and use, including calibration and standardization, field deviations, and sampling limitations, will be recorded on the daily field log. Field records will be initialed by the reviewer prior to their incorporation into reports or use in making program decisions. Changes or corrections to field form entries will be completed by striking out the incorrect entry with a single line and initialing (by the person making the correction) and dating the correction. The original item, although erroneous, must remain legible beneath the crossed-out line. The new information should be written clearly above the crossed-out item.

Examples of the field forms listed below are found in Attachment 1:

- Daily report/field log
- Chain of custody form
- Groundwater sampling form
- Soil gas sampling form
- Groundwater monitoring well gauging form

14.15 Sampling Quality Control

Quality control (QC) samples will be collected at a frequency designated in Worksheets #12, #20, and #28 of this SAP. QC samples will include field duplicates, matrix spikes (MS), matrix spike duplicates (MSD), equipment blanks, source water blanks, and temperature blanks. If re-usable sampling equipment is used, an equipment blank will be collected from decontaminated sampling equipment. Analytical methods will include performing initial calibrations, continuing calibration, tuning, reagent blanks, instrument blanks, surrogates, replicates, laboratory control spikes, and other applicable QC as defined by the methods.

Samples will be collected, handled, and shipped to the selected laboratory in accordance with the guidelines presented in Worksheet #26 and Worksheet #27, Sample Custody Requirements. Field QC samples and MS/MSD samples will be submitted and analyzed as directed in Worksheet #28. Details of QC sample requirements are further presented in Worksheet #28.

14.16 Data Management Review

Data from this sampling effort will be generated from three primary pathways: field activities, laboratory analytical data, and validated data. Data from all three pathways will be submitted to the Navy Electronic Data Deliverable/Naval Installation Restoration Information Solution (NEDD/NIRIS) website in accordance with the data format described in EWI #6, “Environmental Data Management and Required Electronic Delivery Standards” (NAVFAC SW, 2005).

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Data generated during field activities will be recorded using a field logbook and field forms. The field team lead will review these forms for compliance with QC criteria established in the SAP for completeness and accuracy.

Upon sample arrival, the analytical laboratory will verify each sample's physical condition and ensure that all pertinent documentation associated with each sample is complete. Data generated from the laboratory analysis will be recorded in hardcopy and in electronic data deliverables for submission to the Navy NEDD/NIRIS database. Analytical laboratory staff will verify the data according to the process described in Worksheet #34. The laboratory QA director will review the data before they are submitted for third-party data validation. Details on data validation are provided in the next section and in Worksheets #35–37.

Pertinent data (i.e., geological, spatial, and temporal descriptions) from the field records and third-party-validated electronic data deliverables will then be entered into the Navy's NEDD/NIRIS web-based database. Through the web-based system, the data can be compiled rapidly, plotted in GIS, and reviewed for changes in target analyte concentrations at each sampling point. Hardcopy field records will be stored in a secure project file.

14.17 Third-Party Data Validation

Data generated for this project will be reviewed and verified by the MMEC Group QCM and validated by LDC, an independent, third-party data validation laboratory located in Carlsbad, California. The data validation process and criteria are described in Worksheets #35–37. These requirements are established in accordance with EWI #1, "Chemical Data Validation" (NAVFAC SW, 2001), "Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review" (U.S. EPA, 2014), and the "Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review" (U.S. EPA, 2014).

All data within the data set will be independently validated using the DQOs established for the project. Twenty percent of the data are subjected to a Level IV validation. The remaining 80 percent of the data are validated per Level III procedures. The validator will perform calculation checks for these data and the data for the associated laboratory QC samples. The data validator will also facilitate uploading the validated data into the Navy's NEDD/NIRIS database in accordance with EWI #6 (NAVFAC SW, 2005). The guideline elements for Level III and Level IV data validation are summarized below.

14.17.1 Level III Data Validation

For a Level III data validation effort, the data values for routine and QC samples are generally assumed to be correctly reported by the laboratory. Data quality is assessed by comparing the parameters listed below to the appropriate criteria (or limits) as specified in the project SAP, by Contract Laboratory Program requirements, or by method-specific requirements (e.g., Contract Laboratory Program, SW-846). If calculations for quantitation are verified, they are done on a limited basis and may require raw data in addition to the standard data forms normally present in a data package, including the following:

- Holding times (exceedance)
- Gas chromatograph/mass spectrometer (GC/MS) tune
- Initial and continuing calibrations

SAP WORKSHEET #14 CONTINUED

- Blanks (use of "5X/10X rule" for assessment of contamination)
- Blank spikes/laboratory control samples (LCS)
- Surrogates
- MS/MSD
- Internal standards (IS) area performance

14.17.2 Level IV Data Validation

Level IV data validation follows the U.S. EPA guidelines set forth in the functional guidelines for evaluating organic and inorganic analyses (U.S. EPA, 2008, 2010a). These guidelines apply to analytical data packages that include the raw data (e.g., spectra and chromatograms) and backup documentation for calibration standards, analysis run logs, LCS, dilution factors, and other types of information. This additional information is utilized in the Level IV data validation process for checking calculations of quantified analytical data. Calculations are checked for QC samples (e.g., MS/MSD and LCS data) and routine field samples (including duplicates, and field and equipment rinsate blanks). To ensure that detection limit and data values are appropriate, an evaluation is made of the instrument performance, method of calibration, and original data for calibration standards.

Analytical data may be qualified based on data validation reviews. Qualifiers will be consistent with the applicable U.S. EPA functional guidelines and will be used to provide data users with an estimate of the level of uncertainty associated with the qualified results. The project team will determine the data usability based on data validation results with respect to the following qualifiers:

U – Not detected at or above the stated limit

J – Estimated concentration

R – Non-usable data

UJ – Not detected at or above an estimated sample detection limit

N – Tentative identification

For any instances where the validation qualifiers impact the overall data interpretation and project recommendations, the data usability assessment will discuss the issue and the necessary corrective action.

SAP WORKSHEET #15 REFERENCE LIMITS AND EVALUATION TABLES

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Total Petroleum Hydrocarbons by U.S. EPA 8015B
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
TPH Gasoline (C6-C12)	8006-61-9	See note [2]	See note [2]	100	50	25	15	10	30	100
TPH Jet Fuel (C10-C12)	HZ0600-26-T	See note [2]	See note [2]	500	50	50	25	TBD	TBD	TBD
TPH Diesel (C12-C24)	68834-30-5	See note [2]	See note [2]	500	50	40	16	17	50	500
TPH Motor Oil (C24-C36)	68476-77-7	See note [2]	See note [2]	500	500	400	166	23	50	500

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
DL: Detection Limit
µg/L: milligrams per liter
TBD: To be determined
LOQ: Limit of Quantitation
LOD: Limit of Detection
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Volatile Organic Compounds by U.S. EPA 8260B
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Benzene	71-43-2	See note [2]	See note [2]	1	1	0.4	0.13	0.064	0.25	1
Bromobenzene	108-86-1	See note [2]	See note [2]	1	1	0.4	0.18	0.076	0.25	1
Bromochloromethane	74-97-5	See note [2]	See note [2]	1	1	0.4	0.14	0.13	0.5	1
Bromodichloromethane	75-27-4	See note [2]	See note [2]	1	1	0.4	0.14	0.088	0.25	1
Bromoform	75-25-2	See note [2]	See note [2]	1	1	0.4	0.1	0.17	0.25	1
Bromomethane	74-83-9	See note [2]	See note [2]	2	1	0.8	0.29	0.25	0.5	2
n-Butylbenzene	104-51-8	See note [2]	See note [2]	1	1	0.4	0.12	0.25	0.5	1
sec- Butylbenzene	135-98-8	See note [2]	See note [2]	1	1	0.4	0.12	0.086	0.25	1
tert- Butylbenzene	98-06-6	See note [2]	See note [2]	1	1	0.4	0.14	0.11	0.25	1
Carbon tetrachloride	56-23-5	See note [2]	See note [2]	0.14	1	0.4	0.15	0.12	0.25	1
Chlorobenzene	108-90-7	See note [2]	See note [2]	23.3	1	0.4	0.12	0.15	0.25	2
Chloroethane	75-00-3	See note [2]	See note [2]	2	1	0.8	0.34	0.099	0.25	2
Chloroform	67-66-3	See note [2]	See note [2]	1	1	0.4	0.12	0.1	0.25	1
Chloromethane	74-87-3	See note [2]	See note [2]	2	1	0.8	0.25	0.077	0.25	2
2-Chlorotoluene	95-49-8	See note [2]	See note [2]	1	1	0.4	0.15	0.078	0.25	1
4-Chlorotoluene	106-43-4	See note [2]	See note [2]	1	1	0.4	0.15	0.081	0.25	1
Dibromochloromethane	124-48-1	See note [2]	See note [2]	33	1	0.4	0.13	0.13	0.25	1
Dibromo-3-chloropropane	96-12-8	See note [2]	See note [2]	0.54	2	0.8	0.32	0.41	0.5	1

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Volatile Organic Compounds by U.S. EPA 8260B
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Dibromomethane	74-95-3	See note [2]	See note [2]	10	1	0.8	0.21	0.21	0.5	2
Dichlorodifluoromethane (Freon 12)	75-71-8	See note [2]	See note [2]	2	1	0.4	0.16	0.084	0.25	2
Dichloromethane (Methylene Chloride)	75-09-2	See note [2]	See note [2]	10	1	0.8	0.35	0.27	0.5	1
1,2-Dichlorobenzene (DCB)	95-50-1	See note [2]	See note [2]	200	1	0.4	0.14	0.06	0.25	1
1,2-Dichloroethane	107-06-2	See note [2]	See note [2]	0.5	1	0.8	0.22	0.1	0.25	1
1,3-DCB	541-73-1	See note [2]	See note [2]	1	1	0.4	0.11	0.078	0.25	1
1,2-Dichloropropane	78-87-5	See note [2]	See note [2]	1.7	1	0.4	0.15	0.097	0.25	1
1,3-Dichloropropene	542-75-6	See note [2]	See note [2]	0.5	1	0.8	0.22	0.072	0.25	1
1,4-DCB	106-46-7	See note [2]	See note [2]	1.7	1	0	0.13	0.12	0.25	1
2,2-Dichloropropane	594-20-7	See note [2]	See note [2]	1	1	0.4	0.13	0.14	0.25	1
1,1-Dichloroethane	75-34-3	See note [2]	See note [2]	1.7	1	0.4	0.15	0.068	0.25	1
1,1-Dichloroethene (DCE)	75-35-4	See note [2]	See note [2]	2	1	0.4	0.14	0.083	0.25	1
1,1-Dichloropropene	563-58-6	See note [2]	See note [2]	1	1	0.4	0.14	0.079	0.25	1
trans-1,2-DCE	156-60-5	See note [2]	See note [2]	3.3	1	0.4	0.11	0.083	0.25	1
cis-1,2-DCE	156-59-2	See note [2]	See note [2]	2	1	0.4	0.1	0.087	0.25	1
Di-Isopropyl Ether	108-20-3	See note [2]	See note [2]	10				0.25	0.5	2
Ethylbenzene	100-41-4	See note [2]	See note [2]	100	1	0.4	0.15	0.086	0.25	1

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Volatile Organic Compounds by U.S. EPA 8260B
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Ethylene Dibromide (EDB)	106-93-4	See note [2]	See note [2]	0.14	2	0.8	0.22	0.13	0.5	1
Ethyl Tertiary Butyl Ether	637-92-3	See note [2]	See note [2]	10	2	0.40	0.15	0.43	1	1
Hexachlorobutadiene	87-68-3	See note [2]	See note [2]	1	1	0.80	0.22	0.087	0.25	1
Isopropylbenzene (cumene)	98-82-8	See note [2]	See note [2]	1	1	0.4	0.12	0.083	0.25	1
4-Isopropyltoluene	99-87-6	See note [2]	See note [2]	1	1	0.4	0.13	0.085	0.25	1
Methyl tert-butyl ether (MTBE)	1634-04-4	See note [2]	See note [2]	14.3	2	0.4	0.19	0.11	0.25	2
Naphthalene	91-20-3	See note [2]	See note [2]	2	1	0.40	0.15	0.16	0.25	5
n-Propylbenzene	103-65-1	See note [2]	See note [2]	1	1	0.4	0.15	0.062	0.25	1
Styrene	100-42-5	See note [2]	See note [2]	33	1	0.4	0.15	0.074	0.25	1
Tertiary Amyl Methyl Ether	994-05-8	See note [2]	See note [2]	10	2	0.40	0.15	0.36	1	2
Tertiary Butyl Alcohol	75-65-0	See note [2]	See note [2]	10	50	10.00	4.25	5	10	10
Toluene	108-88-3	See note [2]	See note [2]	200	1	0.8	0.25	0.072	0.25	1
Trichloroethene (TCE)	79-01-6	See note [2]	See note [2]	50	1	0.4	0.13	0.25	0.5	1
Trichlorofluoromethane	75-69-4	See note [2]	See note [2]	1.7	1	0.8	0.23	0.11	0.25	1
1,1,1,2-Tetrachloroethane	630-20-6	See note [2]	See note [2]	50	1	0.4	0.1	0.099	0.25	1
1,1,1-Trichloroethane	71-55-6	See note [2]	See note [2]	1	1	0.4	0.19	0.069	0.25	1

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Volatile Organic Compounds by U.S. EPA 8260B
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
1,1,2,2-Tetrachloroethane	79-34-5	See note [2]	See note [2]	66	1	0.4	0.15	0.098	0.25	1
Tetrachloroethene (PCE)	127-18-4	See note [2]	See note [2]	1	1	0.4	0.15	0.18	0.5	1
1,1,2-Trichloroethane	79-00-5	See note [2]	See note [2]	1.7	1	0.8	0.31	0.15	0.25	1
1,2,3-Trichlorobenzene	87-61-6	See note [2]	See note [2]	1.7	1	0.4	0.14	0.087	0.25	2
1,2,3-Trichloropropane	96-18-4	See note [2]	See note [2]	1	1	0.4	0.13	0.15	0.25	1
1,3,5-Trimethylbenzene	108-67-8	See note [2]	See note [2]	1	1	0.40	0.14	0.081	0.25	1
1,2,4-Trichlorobenzene	120-82-1	See note [2]	See note [2]	1	1	0.4	0.1	0.078	0.25	5
1,2,4-Trimethylbenzene	95-63-6	See note [2]	See note [2]	1.7	1	0.40	0.12	0.084	0.25	1
Xylenes (m&p-Xylenes, o-Xylene)	1330-20-7	See note [2]	See note [2]	1	1	0.4	0.18	0.2	0.5	3
Vinyl chloride	75-01-4	See note [2]	See note [2]	0.14	1	0.8	0.22	0.084	0.25	2
Acetone	67-64-1	See note [2]	See note [2]	10	10	5.0	2.1	0.34	1	2
Carbon Disulfide	75-15-0	See note [2]	See note [2]	1	2	0.4	0.16	0.051	0.25	2
2-Butanone (Methyl Ethyl Ketone)	78-93-3	See note [2]	See note [2]	10	2	0.8	0.35	0.52	1	5

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.
NA: not applicable to this project
DL: Detection Limit
µg/L: milligrams per liter
LOQ: Limit of Quantitation
LOD: Limit of Detection
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Semi-Volatile Organic Compounds by U.S. EPA 8270C
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Bis(2-chlorethyl)ether	111-44-4	See note [2]	See note [2]	10	10	3.0	1.5	1	3	10
2-Chlorophenol	95-57-8	See note [2]	See note [2]	10	10	6.0	1.6	1	3	10
1,3-Dichlorobenzene	541-73-1	See note [2]	See note [2]	10	10	3.0	1.4	1	3	10
1,4-Dichlorobenzene	106-46-7	See note [2]	See note [2]	1.2	10	3.0	1.4	1	3	10
1,2-Dichlorobenzene	95-50-1	See note [2]	See note [2]	200	10	3.0	1.5	1	3	10
2-Methylphenol	95-48-7	See note [2]	See note [2]	10	10	3.0	0.93	1	3	10
Bis(2-chloroisopropyl) ether	108-60-1	See note [2]	See note [2]	10	10	3.0	1.3	1	3	10
3- & 4-Methylphenol	65794-96-9	See note [2]	See note [2]	10	30	3	1.15	2	3	10
N-Nitroso-di-n-Propylamine	621-64-7	See note [2]	See note [2]	10	10	3.0	1.4	1.5	3	10
Hexachloroethane	67-72-1	See note [2]	See note [2]	10	10	3.0	1.4	1	3	10
Nitrobenzene	98-95-3	See note [2]	See note [2]	10	10	6.0	1.6	1	3	10
Isophorone	78-59-1	See note [2]	See note [2]	10	10	3.0	1	1	3	10
2-Nitrophenol	88-75-5	See note [2]	See note [2]	20	10	6.0	1.9	1.5	3	10
2,4-Dimethylphenol	105-67-9	See note [2]	See note [2]	10	10	6.0	2.2	1	3	10
Bis (2-chlorethoxy) Methane	111-91-1	See note [2]	See note [2]	10	10	3.0	1	1	3	10
2,4-Dichlorophenol	120-83-2	See note [2]	See note [2]	10	10	6.0	2.6	1	3	10
1,2,4-Trichlorobenzene	120-82-1	See note [2]	See note [2]	10	10	3.0	1.4	1	3	10
Naphthalene	91-20-3	See note [2]	See note [2]	10	10	3.0	1.3	1	3	10

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Semi-Volatile Organic Compounds by U.S. EPA 8270C
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
4-Chloroaniline	106-47-8	See note [2]	See note [2]	10	10	6.0	2	2	3	10
Hexachlorobutadiene	87-68-3	See note [2]	See note [2]	10	10	3.0	1.3	1	3	10
4-Chloro-3-Methylphenol	59-50-7	See note [2]	See note [2]	10	10	6.0	2	1	3	10
2-Methylnaphthalene	91-57-6	See note [2]	See note [2]	10	10	3.0	1.5	1	3	10
Hexachlorocyclo-pentadiene	77-47-4	See note [2]	See note [2]	4.2	50	10.0	5	1	3	10
2,4,6-Trichlorophenol	88-06-2	See note [2]	See note [2]	10	10	6.0	2	1	3	10
2,4,5-Trichlorophenol	95-95-4	See note [2]	See note [2]	10	10	6.0	2	1	3	10
2-Chloronaphthalene	91-58-7	See note [2]	See note [2]	10	10	3.0	1.3	1	3	10
2-Nitroaniline	88-74-4	See note [2]	See note [2]	20	50	6.0	2	1.1	3	10
Dimethyl Phthalate	131-11-3	See note [2]	See note [2]	10	10	3.0	0.88	1	3	10
Acenaphthylene	208-96-8	See note [2]	See note [2]	10	10	3.0	1.1	1	3	10
3-Nitroaniline	99-09-2	See note [2]	See note [2]	20	50	3.0	1.4	1	10	10
Acenaphthene	83-32-9	See note [2]	See note [2]	10	10	3.0	1.1	1	3	10
2,4-Dinitrophenol	51-28-5	See note [2]	See note [2]	50	75	50	20	2	10	50
4-Nitrophenol	100-02-7	See note [2]	See note [2]	20	60	20	6.1	2	10	25
Dibenzofuran	132-64-9	See note [2]	See note [2]	10	10	3.0	1.1	1	3	10
2,4-Dinitrotoluene	121-14-2	See note [2]	See note [2]	10	10	6.0	2	1	3	10
2,6-Dinitrotoluene	606-20-2	See note [2]	See note [2]	10	10	6.0	2	2.2	3	10

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Semi-Volatile Organic Compounds by U.S. EPA 8270C
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Diethyl Phthalate	84-66-2	See note [2]	See note [2]	10	10	3.0	0.93	1	3	10
4-Chlorophenyl Phenyl Ether	7005-72-3	See note [2]	See note [2]	10	10	3.0	1.1	1	3	10
Fluorene	86-73-7	See note [2]	See note [2]	10	10	3.0	0.93	1	3	10
4-Nitroaniline	100-01-6	See note [2]	See note [2]	20	50	3.0	1.5	1	3	10
2-Methyl-4,6-Dinitrophenol	8071-51-0	See note [2]	See note [2]	20	60	6	2.2	1.3	3	10
N-Nitrosodiphenylamine	86-30-6	See note [2]	See note [2]	10	10	3.0	0.8	1	3	10
4-Bromophenyl Phenyl Ether	101-55-3	See note [2]	See note [2]	10	10	3.0	1.1	1	3	10
Hexachlorobenzene	118-74-1	See note [2]	See note [2]	1	10	3.0	1.4	1	3	10
Pentachlorophenol	87-86-5	See note [2]	See note [2]	2	60	20	5	1.3	3	50
N-Nitrosodimethylamine	62-75-9	See note [2]	See note [2]	10	10	3.0	0.96	2	3	10
Phenol	108-95-2	See note [2]	See note [2]	10	10	3.0	1.1	2	3	15
Phenanthrene	85-01-8	See note [2]	See note [2]	10	10	3.0	1	1	3	10
Anthracene	120-12-7	See note [2]	See note [2]	10	10	3.0	1	1	3	10
Di-n-Butyl Phthalate	84-74-2	See note [2]	See note [2]	10	10	3.0	1.1	1	3	10
Fluoranthene	206-44-0	See note [2]	See note [2]	10	10	3.0	0.8	1	3	10
Pyrene	129-00-0	See note [2]	See note [2]	10	10	3.0	1.4	1	3	10
Butyl Benzyl Phthalate	85-68-7	See note [2]	See note [2]	10	10	3.0	1.4	1	3	10
3,3'-Dichlorobenzidine	91-94-1	See note [2]	See note [2]	50	50	3.0	0.96	1.3	3	50

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Semi-Volatile Organic Compounds by U.S. EPA 8270C
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific (non-rad-impacted wells)			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Benz(a)anthracene	56-55-3	See note [2]	See note [2]	10	10	3.0	1	1	3	10
Bis(2-ethylhexyl) Phthalate	117-81-7	See note [2]	See note [2]	1.3	10	3.0	1	1.9	3	10
Chrysene	218-01-9	See note [2]	See note [2]	10	10	3.0	0.8	1	3	10
Di-n-Octyl Phthalate	117-84-0	See note [2]	See note [2]	10	10	3.0	1.5	1	3	15
Benzo(b)fluoranthene	205-99-2	See note [2]	See note [2]	10	10	3.0	1.2	1	3	10
Benzo(k)fluoranthene	207-08-9	See note [2]	See note [2]	10	10	3.0	0.96	1	3	10
Benzo(a)pyrene	50-32-8	See note [2]	See note [2]	1	10	3.0	0.8	1	3	10
Indeno(1,2,3-cd)pyrene	193-39-5	See note [2]	See note [2]	10	15	10	3.4	1	3	10
Dibenz(a,h)anthracene	53-70-3	See note [2]	See note [2]	10	10	6.0	2	1	3	10
Benzo(g,h,i)perylene	191-24-2	See note [2]	See note [2]	10	10	3.0	1.4	1	3	10
Benzoic acid	65-85-0	See note [2]	See note [2]	50	75	56	20	5	10	50

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
DL: Detection Limit
LOD: Limit of Detection
µg/L: milligrams per liter
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Polycyclic Aromatic Hydrocarbons by U.S. EPA 8270SIM
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Acenaphthene	83-32-9	See note [2]	See note [2]	5	0.050	0.015	0.005	0.035	0.1	0.2
Acenaphthylene	208-96-8	See note [2]	See note [2]	5	0.050	0.015	0.005	0.038	0.1	0.2
Anthracene	120-12-7	See note [2]	See note [2]	1.5	0.050	0.015	0.0044	0.039	0.1	0.2
Benzo(a)anthracene	56-55-3	See note [2]	See note [2]	1	0.050	0.015	0.0046	0.031	0.1	0.2
Benzo(a)pyrene	50-32-8	See note [2]	See note [2]	0.078	0.050	0.015	0.0044	0.053	0.1	0.2
Benzo(b)fluoranthene	205-99-2	See note [2]	See note [2]	1	0.050	0.030	0.012	0.055	0.1	0.2
Benzo(g,h,i)perylene	191-24-2	See note [2]	See note [2]	1	0.050	0.015	0.0055	0.04	0.1	0.2
Benzo(k)fluoranthene	207-08-9	See note [2]	See note [2]	1	0.050	0.030	0.0078	0.073	0.1	0.2
Chrysene	218-01-9	See note [2]	See note [2]	1	0.050	0.015	0.0040	0.039	0.1	0.2
Dibenz(a,h,)anthracene	53-70-3	See note [2]	See note [2]	1	0.050	0.030	0.015	0.046	0.1	0.2
Fluoranthene	74-84-0	See note [2]	See note [2]	1	0.050	0.015	0.0043	0.034	0.1	0.2
Fluorene	86-73-7	See note [2]	See note [2]	1	0.050	0.015	0.004	0.032	0.1	0.2
Indeno(1,2,3-cd)pyrene	193-39-5	See note [2]	See note [2]	1	0.050	0.030	0.014	0.04	0.1	0.2
Naphthalene	91-20-3	See note [2]	See note [2]	1	0.050	0.015	0.005	0.068	0.1	0.2
Phenanthrene	85-01-8	See note [2]	See note [2]	1	0.050	0.015	0.0063	0.065	0.1	0.2
Pyrene	129-00-0	See note [2]	See note [2]	1	0.050	0.015	0.0042	0.037	0.1	0.2

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
DL: Detection Limit
LOD: Limit of Detection
µg/L: milligrams per liter
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Organochlorine Pesticides by U.S. EPA 8081A
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Alpha benzene hexachloride (BHC)	319-84-6	See note [2]	See note [2]	0.05	0.05	0.025	0.007	0.01	0.02	0.055
Beta BHC	319-84-6	See note [2]	See note [2]	0.05	0.05	0.025	0.007	0.013	0.02	0.05
Delta BHC	319-86-8	See note [2]	See note [2]	0.05	0.05	0.025	0.011	0.01	0.02	0.05
Gamma BHC (Lindane)	58-89-9	See note [2]	See note [2]	0.067	0.05	0.012	0.006	0.01	0.02	0.05
Heptachlor	76-44-8	See note [2]	See note [2]	0.0086	0.05	0.025	0.007	0.01	0.02	0.1
Aldrin	309-00-2	See note [2]	See note [2]	0.05	0.05	0.012	0.006	0.01	0.02	0.05
Heptachlor epoxide	1024-57-3	See note [2]	See note [2]	0.006	0.05	0.012	0.006	0.017	0.02	0.05
Endosulfan I	959-98-8	See note [2]	See note [2]	0.05	0.05	0.012	0.006	0.01	0.02	0.05
Dieldrin	60-57-1	See note [2]	See note [2]	0.1	0.05	0.025	0.012	0.01	0.02	0.05
4,4'- dichloro-diphenyl-dichloroethylene (DDE)	72-55-9	See note [2]	See note [2]	0.1	0.05	0.025	0.012	0.01	0.02	0.05
Endrin	72-20-8	See note [2]	See note [2]	0.67	0.05	0.025	0.012	0.017	0.02	0.05
Endosulfan II	33213-65-9	See note [2]	See note [2]	0.1	0.05	0.025	0.012	0.01	0.02	0.05
4,4'-dichloro-diphenyl-dichloroethane (DDD)	72-54-8	See note [2]	See note [2]	0.1	0.05	0.025	0.012	0.01	0.02	0.05
Endosulfan sulfate	1031-07-8	See note [2]	See note [2]	0.1	0.05	0.025	0.012	0.01	0.02	0.055
4,4'-dichloro-diphenyl-trichloroethane (DDT)	50-29-3	See note [2]	See note [2]	0.1	0.05	0.025	0.012	0.01	0.02	0.05

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Organochlorine Pesticides by U.S. EPA 8081A
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Endrin ketone	53494-70-5	See note [2]	See note [2]	0.67	0.1	0.08	0.02	0.01	0.02	0.05
Methoxychlor	72-43-5	See note [2]	See note [2]	10	0.1	0.08	0.0418	0.01	0.02	0.1
Endrin aldehyde	7421-93-4	See note [2]	See note [2]	0.67	0.1	0.08	0.025	0.01	0.02	0.05
Chlordane (alpha, gamma isomers)	5103-71-9	See note [2]	See note [2]	0.05	0.05	0.025	0.012	0.01	0.02	0.05
Toxaphene	8001-35-2	See note [2]	See note [2]	2	2	1	0.51	0.27	0.4	2

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
DL: Detection Limit
µg/L: milligrams per liter
LOQ: Limit of Quantitation
LOD: Limit of Detection
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Polychlorinated Biphenyls by U.S. EPA 8082A
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Aroclor 1016	12674-11-2	See note [2]	See note [2]	0.16	1	0.25	0.09	0.35	0.3	1
Aroclor 1221	11104-28-2	See note [2]	See note [2]	0.32	1	0.25	0.11	0.35	0.3	1
Aroclor 1232	11141-16-5	See note [2]	See note [2]	0.14	1	0.50	0.17	0.35	0.3	1
Aroclor 1242	53469-21-9	See note [2]	See note [2]	0.14	1	0.25	0.12	0.35	0.3	1
Aroclor 1248	12672-29-6	See note [2]	See note [2]	0.16	1	0.25	0.10	0.35	0.3	1
Aroclor 1254	11097-69-1	See note [2]	See note [2]	0.169	1	0.25	0.10	0.17	0.3	1
Aroclor 1260	11096-82-5	See note [2]	See note [2]	0.169	1	0.25	0.10	0.17	0.3	1

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
DL: Detection Limit
LOD: Limit of Detection
µg/L: milligrams per liter
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Dissolved Metals by U.S. EPA 6010B/6020A/7470A/7196A
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Aluminum	7429-90-5	See note [2]	See note [2]	333	100	60	25	17.3	30	30
Antimony*	7440-36-0	See note [2]	See note [2]	5	6	4	2	1.7	5	5
Arsenic*	7440-38-2	See note [2]	See note [2]	10	5	3	1	1.2	3.5	10
Barium	7440-39-3	See note [2]	See note [2]	333	3	2	0.5	0.22	0.66	2
Beryllium*	7440-41-7	See note [2]	See note [2]	1.3	1	0.5	0.2	0.35	0.5	0.5
Cadmium*	7440-43-9	See note [2]	See note [2]	1.7	1.5	1	0.5	0.1	0.3	0.5
Calcium	7440-70-2	See note [2]	See note [2]	500	500	300	150	68.1	100	100
Chromium* (total)	7440-47-3	See note [2]	See note [2]	17	5	4.5	1.5	1	9.8	10
Chromium (hexavalent) (7196A)	18540-29-9	See note [2]	See note [2]	17	0.010	0.0050	0.0021	2.6	5	10
Cobalt	7440-48-0	See note [2]	See note [2]	2	3	2	1	0.22	0.66	2
Copper*	7440-50-8	See note [2]	See note [2]	433	5	3	1	0.68	1.4	3
Iron	7439-89-6	See note [2]	See note [2]	100	100	50	25	32.8	50	50
Lead*	7439-92-1	See note [2]	See note [2]	5	2	1.2	0.6	0.17	0.51	3
Magnesium	7439-95-4	See note [2]	See note [2]	500	100	50	25	5.6	16.8	50
Manganese	7439-96-5	See note [2]	See note [2]	10	8	5	2	0.88	0.75	2
Mercury* (EPA 7470A)	7439-97-6	See note [2]	See note [2]	0.67	0.25	0.2	0.1	0.06	0.15	0.2

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Dissolved Metals by U.S. EPA 6010B/6020A/7470A/7196A
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Molybdenum	7439-98-7	See note [2]	See note [2]	20	3	2	1	1	3	5
Nickel*	7440-02-0	See note [2]	See note [2]	33	3	2	1	0.8	1.2	5
Potassium	7440-09-7	See note [2]	See note [2]	500	120	100	25	41.6	100	100
Selenium	7782-49-2	See note [2]	See note [2]	17	3	2	1	1.6	4.8	5
Silver*	7440-22-4	See note [2]	See note [2]	5	1	0.6	0.3	0.82	1	2
Sodium	7440-23-5	See note [2]	See note [2]	500	750	500	250	23.7	45	50
Thallium	7440-28-0	See note [2]	See note [2]	0.67	1.5	1	0.5	0.55	1.6	2
Vanadium	7440-62-2	See note [2]	See note [2]	10	20	12	6	2.4	7.1	10
Zinc*	7440-66-6	See note [2]	See note [2]	20	20	12	4	9.3	10	12

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
DL: Detection Limit
LOD: Limit of Detection
µg/L: milligrams per liter
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Anions by U.S. EPA 300.0
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Chloride	16887-00-6	See note [2]	See note [2]	200	1.0	0.1	0.037	20	60	200
Nitrate	14797-55-8	See note [2]	See note [2]	15,000	0.05	0.05	0.022	4	6	20
Nitrite	14797-65-0	See note [2]	See note [2]	333	0.05	0.05	0.016	3	6	20
Sulfate (as SO4)	14808-79-8	See note [2]	See note [2]	500	1.0	0.15	0.049	50	150	500

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
DL: Detection Limit
µg/L: milligrams per liter
LOQ: Limit of Quantitation
LOD: Limit of Detection
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Radionuclides by U.S. EPA 900.0/901.1/903.0/904.0/905.0/906.0/DOE A-01 R modified
Concentration Level: pCi/L

Analyte	CAS Number	Comparison Criteria (pCi/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (pCi/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])	
					LOQs (pCi/L)	LODs (pCi/L)	DLs (pCi/L)	MDAs (pCi/L)	MDLs (pCi/L)
Gross Alpha	12587-46-1	See note [2]	See note [2]	5	NA	NA	NA	3.0	NA
Gross Beta	12587-47-2	See note [2]	See note [2]	3 mrem/yr	NA	NA	NA	4.0	NA
Cesium-137	10045-97-3	See note [2]	See note [2]	20	NA	NA	NA	20.0	NA
Radium-226	13982-63-3	See note [2]	See note [2]	1.7	NA	NA	NA	1.0	NA
Radium-228	15262-20-1	See note [2]	See note [2]	1.7	NA	NA	NA	1.0	NA
Strontium-90	10098-97-2	See note [2]	See note [2]	3	NA	NA	NA	3.0	NA
Tritium	10028-17-8	See note [2]	See note [2]	6,667	NA	NA	NA	500	NA
Uranium-234	13966-29-5	See note [2]	See note [2]	6.7	NA	NA	NA	1.0	NA
Uranium-235	15117-96-1	See note [2]	See note [2]	6.7	NA	NA	NA	1.0	NA
Uranium-238	7440-61-1	See note [2]	See note [2]	6.7	NA	NA	NA	1.0	NA

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
mrem/yr: millirem per year
DL: Detection Limit
LOD: Limit of Detection
pCi/L: picocuries per liter
CAS: Chemical Abstracts Service
MDL: Method Detection Limit
MDA: Minimum Detectable Activity

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Others by various (reference Worksheet #19 for analytical methods)
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Alkalinity	NA	See note [2]	See note [2]	1	5000	4.0	1.4	0.54	1.5	5
Ferrous Iron	NA	See note [2]	See note [2]	NA[3]	NA[3]	NA[3]	NA[3]	NA[3]	NA[3]	NA[3]
Total Dissolved Solids (TDS)	NA	See note [2]	See note [2]	10	10,000	TBD	5,400	3.5	5	5
Cyanide	57-12-5	See note [2]	See note [2]	67	10.0	5.00	2.00	2.9	5	10
1,4-Dioxane	123-91-1	See note [2]	See note [2]	1	1	0.5	0.20	1.2	5	5
Biological Oxygen Demand (BOD)	NA	See note [2]	See note [2]	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Chemical Oxygen Demand (COD)	NA	See note [2]	See note [2]	TBD	50,000	25,000	1,300	TBD	TBD	TBD
Total Organic Carbon (TOC)	NA	See note [2]	See note [2]	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Dissolved Organic Compounds (DOC)	NA	See note [2]	See note [2]	TBD	TBD	TBD	TBD	TBD	TBD	TBD

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Groundwater
Analytical Group: Others by various (reference Worksheet #19 for analytical methods)
Concentration Level: µg/L

Analyte	CAS Number	Comparison Criteria (µg/L)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-Specific			Laboratory-Specific (rad-impacted wells[1])		
					LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)	LOQs (µg/L)	LODs (µg/L)	DLs (µg/L)
Census DNA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Methane	74-82-8	See note [2]	See note [2]	0.005	5.00	0.365	0.218	0.69	1.1	5
Ethane	74-84-0	See note [2]	See note [2]	0.005	5.00	0.680	0.573	1	2	5
Ethene	74-85-1	See note [2]	See note [2]	0.005	5.00	0.635	0.398	0.85	1.9	5
Sulfide	18496-25-8	See note [2]	See note [2]	1000	4000	1250	793	450	1,000	1,000

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
DL: Detection Limit
LOD: Limit of Detection
µg/L: milligrams per liter
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Soil Gas
Analytical Group: Volatile Organic Compounds in Air by U.S. EPA TO-15
Concentration Level: µg/m³

Analyte	CAS Number	Comparison Criteria (µg/m³)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/m³)	Laboratory-Specific		
					LOQs (µg/m³)	LODs (µg/m³)	DLs (µg/m³)
Acetone	67-64-1	See note [2]	See note [2]	1233	12	0.95	0.42
Benzene	71-43-2	See note [2]	See note [2]	NA	1.3	0.64	0.25
Benzyl chloride	100-44-7	See note [2]	See note [2]	TBD	4.1	2.1	0.84
Bromodichloromethane	75-27-4	See note [2]	See note [2]	NA	2.0	1.3	0.44
Bromoform	75-25-2	See note [2]	See note [2]	0.5	4.1	2.1	0.72
Bromomethane	74-83-9	See note [2]	See note [2]	1.7	3.9	3.1	1.30
2-Butanone (MEK)	78-93-3	See note [2]	See note [2]	0.5	2.4	1.2	0.59
1,3-Butadiene	106-99-0	See note [2]	See note [2]	TBD	1.8	0.88	0.34
Carbon disulfide	75-15-0	See note [2]	See note [2]	TBD	2.5	1.2	0.47
Carbon tetrachloride	56-23-5	See note [2]	See note [2]	NA	5.0	3.1	0.94
Chlorobenzene	108-90-7	See note [2]	See note [2]	206	1.4	0.92	0.29
Chloroethane	75-00-3	See note [2]	See note [2]	0.77	2.6	2.1	0.81
Chloroform	67-66-3	See note [2]	See note [2]	NA	1.5	0.97	0.46
Chloromethane	74-87-3	See note [2]	See note [2]	0.5	1.7	0.82	0.41
Dibromochloromethane	124-48-1	See note [2]	See note [2]	NA	3.4	1.7	0.67
1,2-Dibromoethane (EDB)	106-93-4	See note [2]	See note [2]	TBD	6.1	1.5	0.58
1,2-Dichlorobenzene	95-50-1	See note [2]	See note [2]	TBD	4.8	2.4	0.78
1,3-Dichlorobenzene	541-73-1	See note [2]	See note [2]	TBD	4.8	2.4	0.66

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Soil Gas
Analytical Group: Volatile Organic Compounds in Air by U.S. EPA TO-15
Concentration Level: µg/m³

Analyte	CAS Number	Comparison Criteria (µg/m³)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/m³)	Laboratory-Specific		
					LOQs (µg/m³)	LODs (µg/m³)	DLs (µg/m³)
1,4-Dichlorobenzene	106-46-7	See note [2]	See note [2]	TBD	4.8	2.4	0.89
Dichlorodifluoromethane	75-71-8	See note [2]	See note [2]	TBD	4.0	2.0	0.72
1,1-Dichloroethane	75-34-3	See note [2]	See note [2]	0.5	1.2	0.81	0.29
1,2-Dichloroethane	107-06-2	See note [2]	See note [2]	0.02	3.2	0.81	0.36
1,1-Dichloroethene	75-35-4	See note [2]	See note [2]	TBD	3.2	1.6	0.51
cis-1,2-Dichloroethene	156-59-2	See note [2]	See note [2]	12	1.6	0.79	0.35
trans-1,2-Dichloroethene	156-60-5	See note [2]	See note [2]	24	1.6	0.79	0.40
1,2-Dichloropropane	78-87-5	See note [2]	See note [2]	TBD	4.6	3.7	1.1
cis-1,3-Dichloropropene	10061-01-5	See note [2]	See note [2]	TBD	3.6	1.8	0.47
trans-1,3-Dichloropropene	10061-02-6	See note [2]	See note [2]	0.16	1.8	0.91	0.40
Ethylbenzene	100-41-4	See note [2]	See note [2]	366	1.7	0.87	0.27
4-Ethyltoluene	622-96-8	See note [2]	See note [2]	TBD	3.9	2.0	0.92
Hexachlorobutadiene	87-68-3	See note [2]	See note [2]	TBD	21	13	4.6
2-Hexanone	591-78-6	See note [2]	See note [2]	0.5	1.6	0.82	0.36
Methyl-t-Butyl Ether (MTBE)	1634-04-4	See note [2]	See note [2]	1.2	2.9	0.72	0.18
4-Methyl-2-pentanone (MIBK)	108-10-1	See note [2]	See note [2]	0.5	3.3	1.6	0.55
Methylene Chloride	75-09-2	See note [2]	See note [2]	TBD	1.4	0.69	0.25
Styrene	100-42-5	See note [2]	See note [2]	366	1.9	0.96	0.28

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Soil Gas
Analytical Group: Volatile Organic Compounds in Air by U.S. EPA TO-15
Concentration Level: µg/m³

Analyte	CAS Number	Comparison Criteria (µg/m³)	Comparison Criteria Reference	Project Quantitation Limit Goal (µg/m³)	Laboratory-Specific		
					LOQs (µg/m³)	LODs (µg/m³)	DLs (µg/m³)
1,1,2,2-Tetrachloroethane	79-34-5	See note [2]	See note [2]	NA	2.7	1.4	0.47
Tetrachloroethene	127-18-4	See note [2]	See note [2]	1.1	2.7	1.4	0.35
Toluene	108-88-3	See note [2]	See note [2]	133	1.5	0.75	0.19
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	See note [2]	See note [2]	TBD	3.1	3.1	1.3
1,2,4-Trichlorobenzene	120-82-1	See note [2]	See note [2]	TBD	15	8.9	3.2
1,1,1-Trichloroethane	71-55-6	See note [2]	See note [2]	333	1.6	1.1	0.35
1,1,2-Trichloroethane	79-00-5	See note [2]	See note [2]	NA	2.2	1.1	0.36
Trichloroethene	79-01-6	See note [2]	See note [2]	0.37	4.3	2.2	0.56
Trichlorofluoromethane	75-69-4	See note [2]	See note [2]	TBD	4.5	2.2	1.1
1,2,4-Trimethylbenzene	95-63-6	See note [2]	See note [2]	TBD	3.9	2.0	0.79
1,3,5-Trimethylbenzene	108-67-8	See note [2]	See note [2]	TBD	3.9	2.0	0.61
Vinyl acetate	108-05-4	See note [2]	See note [2]	70	2.8	1.4	0.51
Vinyl chloride	75-01-4	See note [2]	See note [2]	0.073	2.0	1.0	0.31
m,p-Xylene	179601-23-1	See note [2]	See note [2]	243	3.5	0.87	0.43
o-Xylene	95-47-6	See note [2]	See note [2]	243	1.7	0.87	0.23

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
DL: Detection Limit
LOD: Limit of Detection
µg/m³: milligrams per liter
CAS: Chemical Abstracts Service

SAP WORKSHEET #15 CONTINUED

Laboratory: TestAmerica
Matrix: Soil Gas
Analytical Group: Fixed Gasses and Methane, Ethane, and Ethene in Air by TO-3
Concentration Level: % v/v

Analyte	CAS Number	Comparison Criteria (% v/v)	Comparison Criteria Reference	Project Quantitation Limit Goal (% v/v)	Laboratory-Specific		
					LOQs (% v/v)	LODs (% v/v)	DLs (% v/v)
Oxygen	7782-44-7	See note [2]	See note [2]	NA	0.2	0.1	0.04
Carbon Dioxide	124-28-9	See note [2]	See note [2]	NA	0.5	0.25	0.1
Carbon Monoxide	630-08-0	See note [2]	See note [2]	NA	0.01	0.003	0.001
Nitrogen	7727-37-9	See note [2]	See note [2]	NA	1	0.2	0.08
Methane	74-82-8	See note [2]	See note [2]	NA	0.5	0.25	0.135
Ethane	74-84-0	See note [2]	See note [2]	NA	1.00E-09	1.00E-09	1.00E-09
Ethene	74-85-1	See note [2]	See note [2]	NA	1.00E-09	1.00E-09	1.00E-09

Notes:

1 Radionuclide-impacted wells are minimal and may include a small set of wells from OU-2A, -2B, -2C, and IR 35
2 Comparison criteria are defined on an OU and IR site specific basis and can be found on Table 17-1. Comparison criteria are not set for all analytes on this worksheet.

NA: not applicable to this project
LOQ: Limit of Quantitation
DL: Detection Limit
LOD: Limit of Detection
% v/v: percent volume
CAS: Chemical Abstracts Service

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SAP WORKSHEET #16 PROJECT SCHEDULE/TIMELINE TABLE (OPTIONAL FORMAT)

The project schedule is presented in a Gantt chart in Figure A-27. The schedule is contingent on the approval of the Sampling and Analysis Plan.

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SAP WORKSHEET #17 SAMPLING DESIGN AND RATIONALE

As noted in Worksheet #10, this SAP has been prepared to demonstrate that the data collected are sufficient to support ongoing CERCLA actions, assist in monitored natural attenuation, provide additional groundwater data related to active treatment systems, and support long-term groundwater monitoring efforts. A second objective is to promote optimization and efficiency in the BGMP. To accomplish the BGMP objectives, this WS#17 describes the sampling design and rationale to implement the BGMP groundwater sampling, groundwater elevation, NAPL monitoring, and soil gas monitoring activities; and present the process to facilitate optimization and efficiency in the BGMP.

17.1 Basewide Groundwater Monitoring Program Tasks

The sampling design and rationale associated with the BGMP elements (groundwater sampling, soil gas monitoring, well installation, hydropunch sampling, surveying, and well decommissioning) are presented below.

17.1.1 Groundwater Sampling

The current OU/IR well network includes over 850 groundwater monitoring wells located across Alameda Point (Figure A-3). Wells shown in this network may or may not be a part of the BGMP, but there is potential these wells will be sampled, repaired, or decommissioned during the implementation of the BGMP. Zoomed-in views of each IR site depicting the wells associated with the site that may be part of the BGMP are shown on Figures A-5 through A-26.

Wells to be sampled will be identified on a quarterly basis as updated SAP tables per the summary provided in Worksheet #18 and will follow groundwater sampling procedures described in more detail in Worksheet #14. An evaluation of the wells to be sampled during each quarterly event will be conducted through collaboration with the Navy and MMEC Group and will depend on CERCLA, LTM, Land Use Control (LUC), and data requirements for each OU/IR site at that time. As stated previously, the primary of the purpose of the BGMP is to monitor groundwater and soil gas conditions at Alameda Point and to provide supplemental data to support proposed and ongoing CERCLA actions (including natural attenuation).

Results for groundwater samples collected as part of the BGMP will be compared with criteria established specifically for each IR site. Remediation goals and comparison criteria, such as those published in the ROD or Proposed Plans for each IR site that may be a part of the BGMP, are provided in Table 17-1.

Table 17-1. Remediation Goals and Comparison Criteria for Each IR Site

OU Number	IR Site Number	Comparison Source	Comparison Criteria Type	Compound	Comparison Criteria Value
Sitewide	Alameda Point	Final Remedial Investigation Report (Tetra Tech EM Inc. 2004)	Background Values - Groundwater	Metals	
				Antimony	37.5 µg/L
				Arsenic	20.7 µg/L
				Barium	570 µg/L
				Beryllium	2.5 µg/L
				Cadmium	3.9 µg/L
				Chromium	12.5 µg/L
				Cobalt	17.2 µg/L
				Copper	24.2 µg/L
				Lead	6.7 µg/L

SAP WORKSHEET #17 CONTINUED

Table 17-1. Remediation Goals and Comparison Criteria for Each IR Site (continued)

OU Number	IR Site Number	Comparison Source	Comparison Criteria Type	Compound	Comparison Criteria Value
				Manganese	1740 µg/L
				Mercury	0.2 µg/L
				Molybdenum	12.7 µg/L
				Nickel	21 µg/L
				Selenium	8.4 µg/L
				Silver	4.9 µg/L
				Thalium	13.8 µg/L
				Vanadium	26.3 µg/L
				Zinc	36.4 µg/L
				Aluminum	1070 µg/L
				Iron	6590 µg/L
				Calcium	157 µg/L
				Magnesium	356 µg/L
				Potassium	147 µg/L
				Sodium	3710 µg/L
OU-1	IR Site 14	Land Use Controls - ROD (Navy 2007c) & Indoor air risk for residential receptors	ICTC	Vinyl Chloride	15 µg/L
	IR Site 16	IR Site 16 ROD ESD (June 2014) Commercial Vapor Inhalation Risk from Groundwater	RG - VI	1,3-dichlorobenzene (DCB)	57,314 µg/L
			RG - VI	1,2-DCE	156,000 µg/L
			RG - VI	1,4-DCB	272 µg/L
			RG - VI	cis-1,2-DCE	13,050 µg/L
			RG - VI	tetrachloroethene (PCE)	1,501 µg/L
			RG - VI	TCE	152 µg/L
			RG - VI	Vinyl Chloride	40 µg/L
	IR Site 6	IR Site 6 SAP (In-situ Bioremediation, June 2014)	RG - MCLs	PCE	5 µg/L
			RG - MCLs	TCE	5 µg/L
			RG - MCLs	cis-1,2-DCE	6 µg/L
			RG - MCLs	Vinyl Chloride	0.5 µg/L
	OU-1 and IR Sites 6,7,8,16	Final Remediation Investigation Report (Tetra Tech EM Inc. 2004)	Background Values	Metals	See Alameda Point Background Values

SAP WORKSHEET #17 CONTINUED

Table 17-1. Remediation Goals and Comparison Criteria for Each IR Site (continued)

OU Number	IR Site Number	Comparison Source	Comparison Criteria Type	Compound	Comparison Criteria Value
OU-2A	IR Site 13	Commercial vapor intrusion (RGs), residential vapor intrusion (ICTC) (Navy 2013) Land Use Controls - ROD (Navy 2012)	RG – VI	Benzene	Residential: 11.26 µg/L Commercial: 37.84 µg/L
			RG - VI	Ethylbenzene	Residential: 31.46 µg/L Commercial: 105.69 µg/L
OU-2B	IR Site 3	Final Proposed Plan & Indoor Vapor Inhalation Risk from Groundwater (Navy 2013)	RG - VI RG - Groundwater Discharges	TCE	116 µg/L in shallow groundwater; 810 µg/L for discharge into Seaplane Lagoon
			RG - VI	Vinyl Chloride	31.4 µg/L
		Final Remedial Investigation Report (Tetra Tech EM Inc. 2004)	Background values	Arsenic	20.7 µg/L
			Background values	Iron	6,585.5 µg/L
	IR Site 4	Final Proposed Plan & Indoor Vapor Inhalation Risk from Groundwater (Navy 2013)	RG - VI RG - Groundwater Discharges	TCE	116 µg/L in shallow groundwater; 810 µg/L for discharge into Seaplane Lagoon
			RG - VI	Vinyl Chloride	31.4 µg/L
	IR Site 11	Final Proposed Plan & Indoor Vapor Inhalation Risk from Groundwater (Navy 2013)	RG for Vapor Intrusion & Groundwater Discharges	TCE	116 µg/L in shallow groundwater; 810 µg/L for discharge into Seaplane Lagoon
			RG for Vapor Intrusion	Vinyl Chloride	31.4 µg/L
	IR Site 21	Final Proposed Plan & Indoor Vapor Inhalation Risk from Groundwater (Navy 2013)	RG – VI RG - Groundwater Discharges	TCE	116 µg/L in shallow groundwater; 810 µg/L for discharge into Seaplane Lagoon
			RG - VI	Vinyl Chloride	31.4 µg/L

SAP WORKSHEET #17 CONTINUED

Table 17-1. Remediation Goals and Comparison Criteria for Each IR Site (continued)

OU Number	IR Site Number	Comparison Source	Comparison Criteria Type	Compound	Comparison Criteria Value
OU-2C	IR Site 5	ROD (Navy BRAC PMO 2014)	RG - First Water Bearing Zone	1,1-dichloroethane (DCA)	1,260 µg/L
			RG - First Water Bearing Zone	TCE	280 µg/L
			RG - First Water Bearing Zone	Vinyl Chloride	75.7 µg/L
			RG - Second Water Bearing Zone	Vinyl Chloride	163 µg/L
			RG - Second Water Bearing Zone	Total VOCs	1,000 µg/L
OU-3	IR Site 1	Occupational & recreational inhalation of VOCs - ROD (Navy 2009)	RG - VI from Groundwater	Vinyl Chloride	6,011 µg/L
		ROD (Navy BRAC PMO 2009) & Historic values of radionuclides (Navy 2009, 2010)	RG - Surface Water	1,1-DCE	3.2 µg/L
			RG - Surface Water	Benzene	71 µg/L
			RG - Surface Water	TCE	81 µg/L
			RG - Surface Water	Vinyl Chloride	525 µg/L
			RG - Surface Water	Bis(2-chlorethyl)ether	1.4 µg/L
			RG - Surface Water	Bis(2-ethylhexyl)phthalate	5.9 µg/L
			RG - Surface Water	2,4-Dimethylphenol	2300 µg/L
	IR Site 1 & IR Site 32	BGMP SAP (AMEC, 2010)	CA MCL*	Gross Alpha	15 pCi/L
			CA MCL*	Gross Beta	4 mrem/year
			CA MCL*	Strontium-90	8 pCi/L
			CA MCL*	Uranium	20 pCi/L
			CA MCL*	Radium 226+228	5 pCi/L
			CA MCL*	Tritium	20,000 pCi/L

SAP WORKSHEET #17 CONTINUED

Table 17-1. Remediation Goals and Comparison Criteria for Each IR Site (continued)

OU Number	IR Site Number	Comparison Source	Comparison Criteria Type	Compound	Comparison Criteria Value
OU-4A	IR Site 2 & VA FED Parcel	ROD (Navy BRAC PMO 2010), TtEC 2013) & Background Values (ROD, Navy 2014) Final Remedial Action Work Plan, Revision 1 (Tetra Tech, 2013)	Background Value	Arsenic	20.7 µg/L
			Background Value	Copper	24.2 µg/L
			Background Value	Nickel	21 µg/L
		(Navy 2010), TtEC 2013)	CTR limit for human consumption of fish	Nickel - surface water	4,600 µg/L
		Final Remedial Action Work Plan, Revision 1 (Tetra Tech, 2013)	CTR limit for human consumption of fish	Pesticides:	
				Aldrin	0.00014 µg/L
				Alpha chlordane	0.0059 µg/L
				a-HCH	0.013 µg/L
				b-HCH	0.046 µg/L
				4,4'-DDE	0.00059 µg/L
				4,4'-DDT	0.00059 µg/L
				Dieldrin	0.00014 µg/L
				Endrin	0.81 µg/L
				Gamma Chlordane	0.00059 µg/L
				Heptachlor	0.00021 µg/L
				Heptachlor Epoxide	0.00011 µg/L
				SVOCs (bis[2-ethylhexyl]phthalate)	5.9 µg/L
OU-5	IR Sites 25, 30, and 31	ROD (Navy 2007) Vapor Risk	RGs - MCLs	Benzene	1 µg/L
			RGs - MCLs	Naphthalene	100 µg/L
OU-6	IR Site 26	ROD, Residential Indoor Air Vapor Risk (Navy PMO 2006)	RGs - MCLs	cis-1,2-DCE	6 µg/L
			RGs - MCLs	TCE	5 µg/L
			RGs - MCLs	Vinyl Chloride	0.5 µg/L
	IR Site 27	ROD (Navy 2008)	RGs - MCLs	1,1-DCA	5 µg/L
			RGs - MCLs	cis-1,2-DCE	6 µg/L
			RGs - MCLs	PCE	5 µg/L
			RGs - MCLs	trans-1,2-DCE	10 µg/L

SAP WORKSHEET #17 CONTINUED

Table 17-1. Remediation Goals and Comparison Criteria for Each IR Site (continued)

OU Number	IR Site Number	Comparison Source	Comparison Criteria Type	Compound	Comparison Criteria Value
			RGs - MCLs	TCE	5 µg/L
			RGs - MCLs	Vinyl Chloride	0.5 µg/L
	IR Site 28	ROD (Navy 2007b) Domestic (residential) use of GW (Navy 2007b)	RGs – Aquatic Receptors	Arsenic	2,000 µg/L
			Background Value	Copper - surface water	3.1 µg/L
No OU Assigned	IR Site 34	ROD (IR Site 34 ROD - April 2011)	MCL (does not apply due to lack of future use of GW- recommended no action for GW)	TCE	5 µg/L
	IR Site 35	RI/FS IR Site 35 - 2007	MCL	Lead	15 µg/L
			ESLs	TPH	100 µg/L
			California Toxics Rule, National Recommended Water Quality Criteria	Other	See CTR for values

Sources:

AMEC 2010 – Sample and Analysis Plan, Basewide Groundwater Monitoring Program, August
Tetra Tech 2013 - Remedial Action Work Plan, Revision 1, November
Tetra Tech 2004 - Final Remedial Investigation Report, OU-1, Sites 6, 7, 8, and 16, November 18.
Navy 2006 - Final ROD, Site 26, August 23.
Navy 2007a - Final ROD OU-1, Sites 6, 7, 8, and 16, September
Navy 2007b - Final ROD, Site 28, September
Navy 2007c - Final ROD Site 14, January 31
Navy 2008 - Final ROD IR Site 27, Dock Zone, February
Navy 2009 - Final ROD IR Site 1 1943-1956 Disposal Area, November
Navy 2010 - IR Site 2, June
Navy 2013 - Proposed Plan for OU-2B, IR Sites 3, 4, 11, and 21, April
Navy 2014 - Final ROD, OU-2C, IR Sites 5, 10, and 12, April
Navy 2007 - Final ROD OU-5/FISCA 2
Navy 2011 - Final ROD IR Site 34, April
Navy 2011 - Final ROD IR Site 35 and Areas of Concern in Transfer Parcel EDC-35, February 16
Navy 2014 - Final Explanation of Significant Difference for Record of Decision for Groundwater at Operable Unit 1, Installation Restoration Site 16, June 2014

Note:

CA MCL* = California Maximum Contaminat Level (MCL). These values are used for comparison purposes only and do not represent remediation goals for the compounds.

17.1.2 Soil Gas Monitoring

The current BGMP network includes soil gas probes, primarily at IR Site 2. Soil gas samples are being collected to monitor the nature and extent of landfill-related contaminants and to meet the DQOs to support remedial efforts (Worksheet #11). The soil gas probes subject to BGMP sampling are shown on Figure A-26.

Annual soil gas monitoring will be conducted during the spring annual event. The soil gas samples will be collected in 1-liter Summa canisters. The purge rate will not exceed 200 mL/min. Vapors in each gas probe will be initially purged a minimum of three casing volumes using a vacuum pump. Soil gas conditions will be measured in the field using a portable soil gas monitor (GEM-5000 or equivalent). The soil gas monitoring will include methane (percent and LEL), oxygen (percent), carbon dioxide (percent), balance gas (percent, assumed to be nitrogen), and hydrogen sulfide (parts per million).

Procedures for soil gas sampling are described in more detail in Worksheet #14. The soil gas sampling measurements will be recorded on the appropriate field forms (Attachment 1).

17.1.3 Well Installation

Well installation activities may be required at multiple OUs and/or IR sites as part of the BGMP. The wells will be installed at locations identified by the Navy in collaboration with MMEC Group. A well may be installed to replace an existing monitoring well that was damaged, provide an extra well for the IR site well network to monitor plume conditions, or to provide additional site information needed for site characterization if needed.

17.1.4 Hydropunch Sampling

Hydropunch sampling is anticipated to take place at various IR sites in support of applicable ROD and NFA recommendations or other data collection needs, or in IR site areas where groundwater wells may not exist. The Navy, in collaboration with the MMEC Group, will identify locations and IR sites where hydropunch sampling is needed through other planning documents or through evaluation of groundwater data collected as part of the BGMP.

17.1.5 Well Maintenance

Well maintenance will be performed on wells identified as being out of compliance with the California Water Resources Department Bulletin 74-90 and/or other local regulations regarding conditions of wells. Minor maintenance of the well structures includes replacing J-plugs, well locks, well box bolts, well box lids, or rubber gaskets. Minor repairs may be conducted at the time of observed deficiencies in the well structure. Major well maintenance includes repair or replacement of entire well boxes, well pads, well monuments, PVC well casing, and/or bollards.

17.1.6 Surveying

Surveying activities may to take place at various OUs and IR sites, generally in conjunction with hydropunch sampling, well installation, or major well repair activities. If a well has been altered or installed, the well box and wellhead will be surveyed by a state-licensed land surveyor. The well components will be surveyed to a known datum for northings and eastings and to mean sea level (msl) for elevation.

SAP WORKSHEET #17 CONTINUED

17.1.7 Well Decommissioning

Well decommissioning at Alameda Point is necessary to remove wells when they are no longer needed or required. This includes wells which monitor a plume that has a reducing trend with a defined footprint where the plume configuration has not changed; COCs have not been detected above RGs or above comparison criteria for at a minimum of two years previously; damaged or unusable wells; and wells that are no longer needed at the site based on remedial actions which have reduced contaminant concentrations (the success of which is evaluated under separate reports and task orders). Well decommissioning also reduces Navy environmental liabilities at Alameda Point; wells may be decommissioned at the discretion of the Navy. Wells that are candidates for decommissioning will be identified on the basis of the optimization analysis, site history, other investigations conducted by the Navy, future site use, and well physical conditions (i.e., damaged, etc.). The Navy may decommission wells throughout the duration of the BGMP as the opportunity arises. Wells that are decommissioned will be reported in the annual report.

17.2 Basewide Groundwater Monitoring Program Optimization

As BGMP activities continue and new data are available, it may become necessary to amend the BGMP to meet changing site conditions and optimize the BGMP to support development and/or implementation of final remedies. Guidelines to determine analytes and sampling frequency were presented in the 2010 BGMP SAP (AMEC, 2010) as well as the 2005 Technical Memorandum prepared (ITSI 2005). This SAP provides a dynamic work strategy that allows for updating the target analyte list and sampling frequency, facilitating field sampling methods, and streamlining reporting and regulatory approval process to optimize the BGMP as appropriate.

BGMP optimization recognizes site-specific decision and data needs, and includes feedback from the various RPMs that oversee the OUs/IR sites and regulatory agencies. Based on the procedures outlined below, wells may be added to or removed from the BGMP program at the discretion of the Navy, if the criteria set forth below are met. The processes for BGMP optimization are presented below.

17.2.1 Target Analyte List Evaluation Process

VOCs are the primary constituents of concern found in groundwater at OU and IR Sites at former NAS Alameda. Others include: metals, SVOCs, pesticides, PCBs, PAHs, TPH, and radionuclides. This SAP will allow for the continuing evaluation of the applicability of sampling OU and IR Sites for these analyte groups. Optimization will also include evaluation of individual target analytes to identify chemicals of potential concern (COPCs) which may require continued monitoring, and to eliminate other analytes, as appropriate.

Table 17-2 lists decision rules that will be used to evaluate and identify target analytes for groundwater and soil gas sampling.

Table 17-2. Analyte Decision Rules

Analyte Suite	Decision Criteria	Evaluation Required
VOCs	VOCs present primary concern regarding site reuse plans.	Identify COPCs and degradation products with RPMs. Propose revised target analyte list if OU/IR Site plumes are fully defined and relatively stable. Remove from BGMP if concentrations reported

SAP WORKSHEET #17 CONTINUED

Analyte Suite	Decision Criteria	Evaluation Required
		during past five sampling events are less than comparison criteria established for specific OU/IR site.
1,4-dioxane	Stabilizer added to chlorinated degreaser solvents. California public health goal 1 micrograms per liter (µg/L).	Remove from BGMP if concentrations reported during past five sampling events are less than public health goal.
Metals and radionuclides	Metals and radionuclides commonly are naturally occurring. Background concentrations of metals have been established and a radionuclide study completed.	Compare list of analytes previously detected to background data or radionuclide study. Identify COPCs that are significantly above background/study concentrations for continued sampling; remove analytes consistently equal to or below appropriate criteria.
TPH	TPH analysis reports results by carbon ranges, not by specific analytes. For example, the presence of aromatic hydrocarbons (benzene, toluene, ethylbenzene, and xylenes – BTEX) will show up as TPH gasoline.	Remove from BGMP if concentrations reported during past five sampling events are less than comparison criteria.
SVOCs and pesticides	BGMP sampling for these analyte suites are limited primarily to former landfill areas.	Detection of these various analytes in groundwater is sporadic. Identify COPCs that are consistently above comparison criteria concentrations for continued sampling; remove non-detect analytes or those consistently below comparison criteria concentrations.

Because the amount of historical BGMP data available from the OU/IR Sites, concentration trends can be evaluated using professional judgement and used to implement the decision rules statements above and identify specific target analytes for optimization consideration. However, as the BGMP does not provide comprehensive discussion or interpretations of contaminant distribution at the various OU/IR Sites and comparison of BGMP groundwater results only provides a “screening level” evaluation of analytical results, secondary decision criteria other than those summarized above may need to be considered (e.g. retaining wells to serve as sentry/cross gradient monitoring, up/down gradient monitoring of source areas/plumes, or evaluation of ongoing remedial actions pursuant to site decision documents). The final selection process for target analytes or COPCs will incorporate the BCT input (Navy and regulators) to ensure that data collection and optimization recommendations tie into site decisions and needs. The results of the BGMP optimization process will be discussed and summarized in BGMP annual reports.

17.2.2 Sampling Frequency Optimization Process

Groundwater sampling frequency at most OU/IR Sites occurs semiannually or annually. The only default exception is groundwater sampling for new monitoring wells added to the BGMP network. New monitoring wells are sampled on a quarterly basis for four quarters. Four quarters of analytical and water level data is sufficient to establish concentration trends and evaluate temporal variations. After four quarters of groundwater sampling are completed, the

SAP WORKSHEET #17 CONTINUED

sampling frequency may be changed to match the sampling frequency of other wells in the OU/IR Sites.

The sampling frequency of the BGMP monitoring wells will continue to be evaluated to streamline sampling activities that support selection and implementation of final remedies. BGMP sampling frequency will be evaluated using trend analysis (rate of change). The MMEC Group will perform temporal and spatial trend analysis of concentration data collected during the planned field work. Data collected from the previous five years of monitoring will be used to construct the concentration versus time plots.

Statistical methods may be used to evaluate the groundwater data using well-specific BGMP data points (target analytes) to evaluate concentration trends and variability and identify potential redundancy in the monitoring program. Mann-Kendall and Sen's method are broadly accepted statistical tools to analyze time-series groundwater monitoring data to quantitatively determine if the measured concentrations of a chemical are increasing, decreasing, or stable over time; and could provide supporting line of evidence to recommend or maintain ongoing MNA. In practice, trend lines are fitted to the most recent data points (typically six points) for each target analyte in a monitoring well. A well may have more than one sampling frequency for all its target analytes. For example, a monitoring well with a low trend and low variability for copper would indicate annual sampling for metals. However, if the same well demonstrates high trend and high variability for tetrachloroethene (PCE), it may be sampled quarterly for VOCs.

The sampling frequency for monitoring wells may be changed based on the continual evaluation of newly collected site data. New data for target analytes will be evaluated for trend and variability, as appropriate, and the sampling frequency will be revised accordingly to respond to site conditions. If target analyte concentrations in a monitoring well are stable, the sampling frequency may be decreased. If trend or variability increase, the sampling frequency may increase. The evaluation trend and variability will be specific to each OU/IR Site.

The final output of this sampling frequency optimization process will incorporate decision-makers input to ensure that data collection ties into site decisions. The results of BGMP sampling frequency optimization will be discussed in annual BGMP reports.

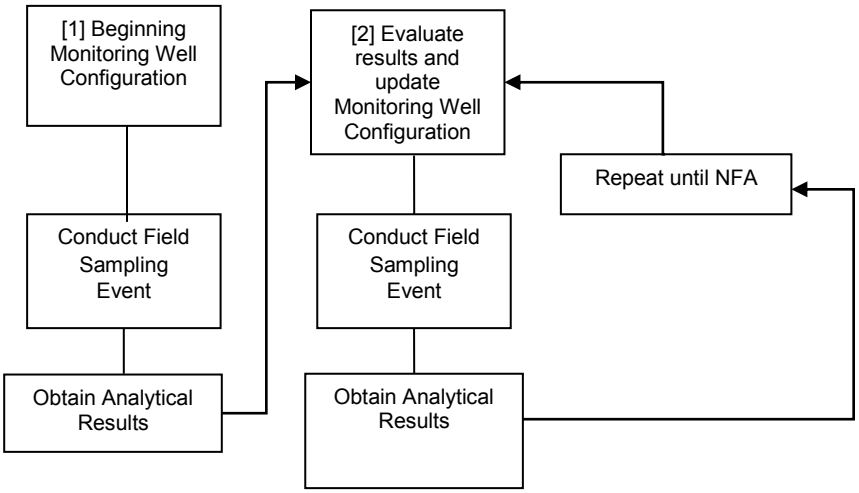
17.3 Basewide Groundwater Monitoring Program Evaluation and Modification

This SAP allows for real-time modification for the BGMP, as appropriate and necessary. The BGMP is designed so it will continually be assessed by the Navy, regulators, and contractors to provide current and relevant data documenting conditions and the various OU/IR Sites.

The dynamic work strategy for updating the monitoring well configuration and analytical requirements, as well as the criteria for making updates, are depicted in the flowcharts provided below. The sampling frequency (e.g., quarterly, semiannually, annually, or in support of CERCLA five-year review) is tailored to the historical characteristics of each well and OU/IR Site, and is continuously re-evaluated using the dynamic work strategy shown below.

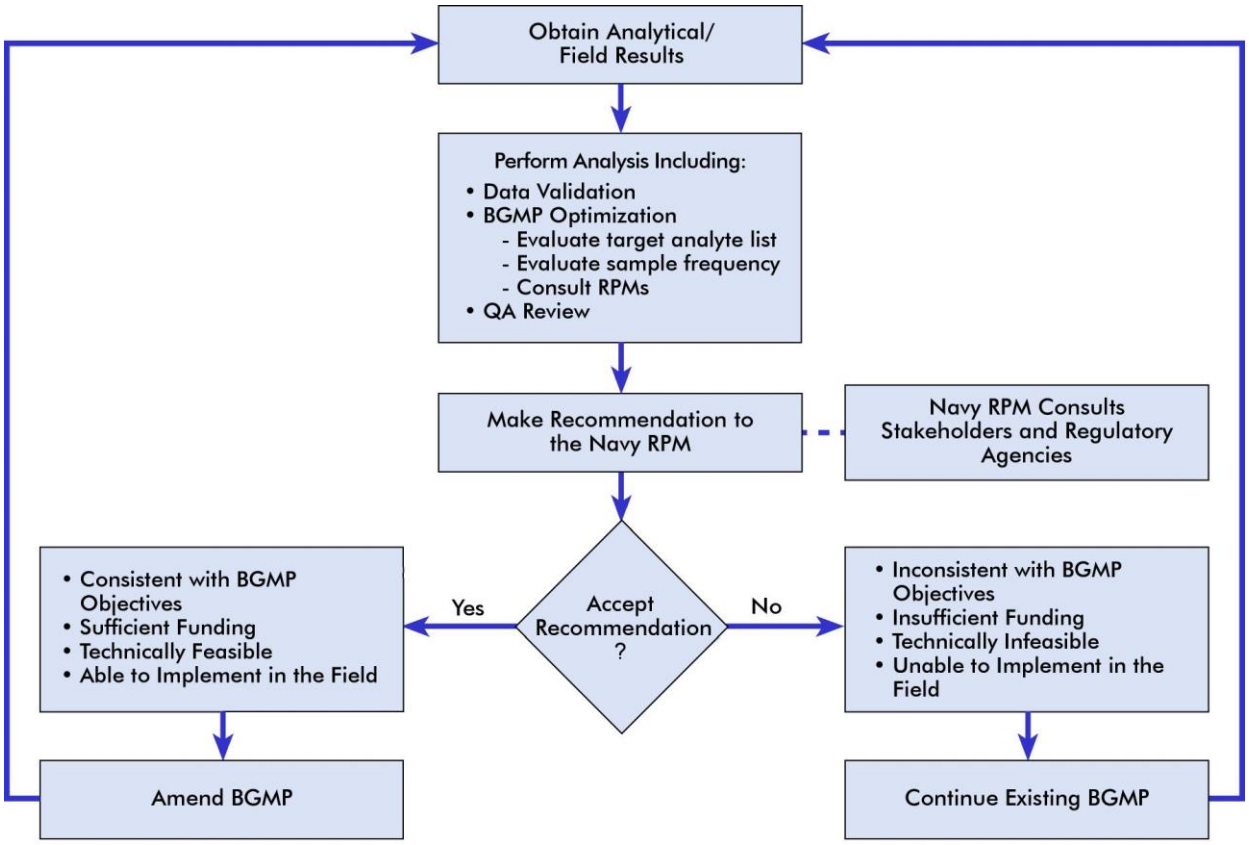
SAP WORKSHEET #17 CONTINUED

Dynamic Work Strategy for Updating the Monitoring Well Configuration



SAP WORKSHEET #17 CONTINUED

Decision-Making Process for Amending BGMP



To facilitate regulatory agency approval of any significant recommended changes, BGMP optimization recommendations will be provided with BGMP Report prepared annually by the Navy. The BGMP optimization changes will be summarized in the annual BGMP reports.

17.4 Basewide Groundwater Monitoring Program Reporting and Agency Approval

As noted above, BGMP optimization changes will be provided in the BGMP annual reports. Information presented in the annual reports will be used to assess whether the monitoring goal/objectives have been achieved at the various OU/IR Sites, or if the data indicate that modification of the BGMP and/or OU/IR Sites are needed to re-evaluate the project objectives.

SAP WORKSHEET #17 CONTINUED

BGMP activities have been conducted since 2002 at the OU/IR Sites, and during this time significant changes have been made by the Navy with input/approval from regulatory agencies. This SAP for the BGMP will be implemented to achieve its goals and objectives, satisfy agency requirements, and provide adequate coverage of groundwater plumes consistent with local, state, and federal regulations. It will also document QA/QC procedures consistent with DQOs. In addition, this SAP provides for BGMP optimization focused towards supporting development and/or implementation of the final remedies. To facilitate agency review and approval of annual BGMP reports, the annual reports will continue to use a similar format to previous reports (streamlined where appropriate, including the removal of groundwater elevation maps) and will include and emphasize plume boundary definitions and on-going remedial efforts. Recommendations for BGMP optimization will be provided in a separate section or an appendix to the annual report.

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SAP WORKSHEET #18 SAMPLING LOCATIONS AND METHODS/SOP REQUIREMENTS TABLE

Wells that are believed to exist at IR sites at Alameda Point and may or may not be sampled, decommissioned, or repaired as part of the Alameda BGMP are provided in the Worksheet #18 table below. This table corresponds with Figure A-3 and Figures A-5 through A-26 which depicts anticipated existing wells at OU/IR sites throughout Alameda Point. The table below provides the planned well sampling frequency and analyticals expected for the implementation of the 2016 BGMP and will be subject to change prior to the implementation of each quarterly sampling event. The anticipated frequency of sampling and wells identified for sampling was developed by utilizing the planned 2015 BGMP sampling network from the *Final Addendum 4 to the Final Sampling and Analysis Plan* (SESTech, 2014a). In addition, information from NIRIS was used to identify the last sampling date and analytical groups sampled at each well, if available. The screened interval, water bearing zone in which the screen is associated, and status of the well (active or inactive) in the last year of BGMP sampling activities completed in 2014 is also displayed. All sampling identified in Worksheet #18 will be conducted in compliance with the sampling procedures and guidelines described in Worksheet #14 of this SAP.

Worksheet #18 will be updated on a quarterly basis prior to each quarterly sampling event. The table below will be modified to show only those wells that will be sampled for that quarterly sampling event and will include all information about the specific analytical groups that will analyzed for each well. The updated Worksheet #18 will be provided to the Navy and other vested parties prior to each quarterly sampling event to ensure the planned well sampling network is documented for each quarterly sampling event and will be used to track that each sampling event is executed as planned.

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SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-1	**IR Site 14**	**Status: Remedial action complete, ISCO and temporary institutional control (OPS, MNA for VOCs, nearing IC termination criterion)**																															
OU-1	IR Site 14	CA02-01	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 14	D14-01	Unknown	SECOND	8/30/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	FS1MJ-MW-1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 14	FS1MJ-MW-2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 14	FS1MJ-MW-4	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 14	M101-A	Unknown	FIRST	7/14/2010	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M101-A-OLD	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 14	M101-C	Unknown	SECOND	8/30/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M112-A	Unknown	FIRST	7/14/2010	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M113-A	Unknown	FIRST	7/13/2010	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M114-A	Unknown	FIRST	8/30/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M14-01	Unknown	FIRST	7/13/2010	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M14-02	Unknown	FIRST	7/14/2010	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M14-03	Unknown	FIRST	7/14/2010	Metals TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M14-04	Unknown	UNDEFINED	7/14/2010	Metals, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M14-05	Unknown	UNDEFINED	7/13/2010	Metals, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M14-06	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active	S							Q	Q	Q	Q					Q										Q

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-1	IR Site 14	M14-07	Unknown	UNDEFINED	7/13/2010	Metals, VOCs, MNA	Inactive																										
OU-1	IR Site 14	M14-08	Unknown	UNDEFINED	7/13/2010	Metals, VOCs	Inactive																										
OU-1	IR Site 14	M14-09D	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q					Q										Q
OU-1	IR Site 14	M14-09S	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q					Q										Q
OU-1	IR Site 14	M14-10	Unknown	UNDEFINED	7/13/2010	Metals, VOCs	Inactive																										
OU-1	IR Site 14	M14-11	Unknown	UNDEFINED	7/14/2010	Metals, VOCs	Inactive																										
OU-1	IR Site 14	M14-12	Unknown	UNDEFINED	7/13/2010	Metals, VOCs	Inactive																										
OU-1	IR Site 14	M14-13	Unknown	UNDEFINED	7/14/2010	Metals, VOCs	Inactive																										
OU-1	IR Site 14	M14-14	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active																										
OU-1	IR Site 14	M14-15	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q					Q										Q
OU-1	IR Site 14	M14-16	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active																										
OU-1	IR Site 14	M14-17	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q					Q										Q
OU-1	IR Site 14	M14-18	Unknown	FWBZ	6/4/2014	Metals, VOCs, MNA	Active																										
OU-1	IR Site 14	M14-19	Unknown	UNDEFINED	7/13/2010	Metals, VOCs	Inactive																										
OU-1	IR Site 14	M14-20D	Unknown	FWBZ	6/4/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q					Q										Q
OU-1	IR Site 14	M14-20S	Unknown	UNDEFINED	7/12/2010	Metals, VOCs	Inactive																										
OU-1	**IR Site 15**	**Status: Recommended for NFA in 2006, Remediation complete and site closed**																															
OU-1	IR Site 15	M15-01	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 15	M15-02	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 15	M15-03	Unknown	FIRST	NA	NA	Inactive																										
OU-1	**IR Site 16**	**Status: Remedial Action via ISCO and in-situ bioremediation (ROD ESD in progress)**																															
OU-1	IR Site 16	608MJ-MW1	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 16	608MJ-MW2	Unknown	FWBZ	5/22/2013	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	608MJ-MW3	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 16	608MW-C2-3	Unknown	UNDEFINED	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-1	IR Site 16	CA09-03	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 16	F16NSMW04	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 16	F16NSMW05	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 16	F16NSMW09	Unknown	FWBZ	5/21/2013	Metals, SVOCs, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	F16SS-MW01	Unknown	FWBZ	NA	NA	Active	S																									S
OU-1	IR Site 16	F16SS-MW03	Unknown	FWBZ	NA	NA	Active	S																									S
OU-1	IR Site 16	F16SS-MW04	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 16	F16SS-MW07	Unknown	FWBZ	NA	NA	Active	S																									S
OU-1	IR Site 16	F16SS-MW08	Unknown	FWBZ	NA	NA	Active	S																									S
OU-1	IR Site 16	M16-04	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 16	MW16-05	5 to 14.5	FIRST	6/16/2009	Metals, PCBs, Pesticides, SVOCs, VOCs, MNA	Inactive																										
OU-1	IR Site 16	MW16-06	Unknown	FIRST	6/17/2009	Metals, PCBs, Pesticides, SVOCs, VOCs, MNA	Inactive																										
OU-1	IR Site 16	MWC2-1	Unknown	FIRST	9/14/2005	Metals, PCBs, SVOCs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 16	MWC2-2	5 to 15	FWBZ	5/22/2013	Metals, PCBs, SVOCs TPH, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	MWC2-3	Unknown	FIRST	6/16/2009	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 16	P-16-IWS-03	Unknown	UNDEFINED	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-1	IR Site 16	P16-MWS05	Unknown	FWBZ	NA	NA	Active	S																									S
OU-1	IR Site 16	P16-MWS05	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 16	S16-MW08	Unknown	SWBZ	5/22/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	S16-MW09	Unknown	SWBZ	5/21/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	S16-MW10	Unknown	SWBZ	5/20/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	S16-MW11	Unknown	SWBZ	5/23/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	S16-MW12	Unknown	SWBZ	5/23/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	S16-MW13	Unknown	SWBZ	5/22/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	S16-TT-MW05	Unknown	SWBZ	5/21/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 16	S16-TT-MW07	Unknown	SWBZ	5/21/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	**IR Site 6**	**Status: Remedial action via ISCO complete and on-going via in-situ bioremediation (ROD in place)**																															
OU-1	IR Site 6	M06-01	Unknown	FWBZ	5/28/2013	Metals, SVOCs, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	M06-02	Unknown	FWBZ	5/28/2013	Metals, SVOCs, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	M06-03	Unknown	FIRST	6/15/2009	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 6	M06-04	Unknown	FIRST	12/10/2007	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-1	IR Site 6	M06-05	Unknown	FIRST	6/16/2009	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 6	M06-06	Unknown	FWBZ	5/24/2013	Metals, SVOCs TPH, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	M06-07	Unknown	FIRST	6/16/2009	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-1	IR Site 6	S6-MW04	Unknown	FWBZ	5/30/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-MW05	Unknown	FWBZ	5/29/2013	Metals, VOCs, MNA	Active	S																									S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-1	IR Site 6	S6-MW06	Unknown	FWBZ	5/23/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-MW07	Unknown	FWBZ	5/29/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-MW08	Unknown	FWBZ	5/24/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-MW09	Unknown	FWBZ	5/24/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-MW10	Unknown	FWBZ	5/23/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-MW11	Unknown	FWBZ	5/30/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-MW12	Unknown	FWBZ	3/13/2013	Metals, VOCs, MNA	Active	S																									S
OU-1	IR Site 6	S6-TT-MW01	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 6	S6-TT-MW02	Unknown	FWBZ	NA	NA	Active	S																									S
OU-1	IR Site 6	S6-TT-MW03	Unknown	SECOND	NA	NA	Inactive																										
OU-1	**IR Site 7**	**Status: Remediation complete under the Alameda Point TPH Program, site closed**																															
OU-1	IR Site 7	CA07-01	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	D07A-01	Unknown	SECOND	9/13/2005	Metals, PAHs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 7	D07A-02	Unknown	SECOND	9/9/2005	Metals, PAHs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 7	M07-10	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	M07-18	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	M07A-03	Unknown	FIRST	3/26/2008	Metals, PAHs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 7	M07A-04	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	M07A-05	Unknown	FIRST	9/13/2005	Metals, PAHs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 7	M07A-06B	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	M07A-07	Unknown	FIRST	9/13/2005	Metals, PAHs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 7	M07A-10	Unknown	FIRST	6/17/2008	Metals, PAHs TPH,	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	**SAMPLING LOCATION**	**SCREEN INTERVAL (FT BGS)**	**WATER-BEARING ZONE**	**LAST YEAR SAMPLED**	**LAST ANALYTICAL GROUPS**	**WELL STATUS IN BGMP**	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						VOCs, MNA																											
OU-1	IR Site 7	S7-B21B	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 7	S7-B28A	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 7	S7-B32	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 7	S7-B33	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 7	S7-B34	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 7	S7-B35	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 7	S7-B37	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-1	IR Site 7	W-1	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	W-2	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	W-3	Unknown	FIRST	NA	NA	Inactive																										
OU-1	**IR Site 8**	**Status: Remediation completed under the Alameda Point TPH Program, site closed**																															
OU-1	IR Site 8	M08-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	**IR Site 13**	**Status: Remedial Action in progress, In-situ bioremediation and MNA, institutional controls in-place (ROD in place, anticipated completion of RA in 2016)**																															
OU-2A	IR Site 13	CA13-01	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	CA13-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	CA13-04	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	CA13-05	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	D13-01	50 to 60	SECOND	3/27/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 13	M07C-06	4 to 14	FIRST	9/8/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 13	M07C-07	3.5 to 13.5	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	M07C-09	Unknown	FIRST	9/8/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	*IR Site 13*	*M13-06*	2 to 9.75	*Shallow*	5/27/2014	Metals TPH, VOCs, MNA	Active																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2A	*IR Site 13*	*M13-07*	2.5 to 12.5	*Shallow*	5/27/2014	Metals TPH, VOCs, MNA	Active																										
OU-2A	*IR Site 13*	*M13-08*	22 to 32	*Shallow*	5/27/2014	Metals TPH, VOCs, MNA	Active																										
OU-2A	*IR Site 13*	*M13-09*	2.5 to 12.5	*Shallow*	5/30/2014	Metals TPH, VOCs, MNA	Active																										
OU-2A	IR Site 13	M13-P	10 to 35	FIRST	9/1/2009	VOCs	Inactive																										
OU-2A	IR Site 13	M-IMF-01	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 13	M-IMF-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	MW-001	5 to 15	FIRST	9/8/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 13	MWOR-1	5 to 15	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	MWOR-2	5 to 15	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	MWOR-3	5 to 15	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 13	MWOR-4	5 to 15	FIRST	9/30/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 13	P13-01	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 13	P13-02	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 13	P13-03	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	*IR Site 13*	*S13-TT-MW01*	5 to 15	*Shallow*	6/9/2014	Metals, VOCs, MNA	Active																										
OU-2A	**IR Site 19**	**Status: Institutional controls in-place (no GW monitoring required)**																															
OU-2A	IR Site 19	372-MW2	2.6 to 12.6	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 19	D19-01	50 to 60	SECOND	3/27/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 19	M19-05	20 to 30	SECOND	NA	NA	Inactive																										
OU-2A	IR Site 19	MWD13-1	26 to 36	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 19	MWD13-2	26.5 to 36.5	FIRST	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2A	IR Site 19	MWD13-3	5 to 15	FIRST	9/30/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 19	MWD-13-4	5 to 15	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 19	S19-TT-MW01	5 to 10	FIRST	8/21/2013	Metals, VOCs, MNA	Inactive																										
OU-2A	**IR Site 22**	**Status: Remedial action complete and site closed**																															
OU-2A	IR Site 22	CAA4C-DGS-PZ01	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 22	D07C-01	49 to 59	SECOND	3/27/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 22	M07C-08	3.5 to 13.5	FIRST	9/30/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 22	MW547-1	3 to 13	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 22	MW547-2	21 to 33	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 22	MW547-3	5 to 15	FIRST	9/8/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 22	MW547-4	5 to 15	FIRST	3/19/2014	Metals, PAHs TPH, VOCs	Inactive																										
OU-2A	IR Site 22	MW-547-5	5 to 15	FIRST	NA	NA	Inactive																										
OU-2A	**IR Site 23**	**Status: Remedial action complete through Alameda Point TPH Program, site closed**																															
OU-2A	IR Site 23	530-MJ-MW-1	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 23	D10B-01	50 to 60	SECOND	NA	NA	Inactive																										
OU-2A	IR Site 23	D10B-02	50 to 60	SECOND	3/27/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 23	M09-05	3.5 to 13.5	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 23	M09-06	4 to 14	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 23	M10B-01	3 to 11	FIRST	9/7/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 23	MW530-1	5 to 15	FIRST	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2A	IR Site 23	MW530-2	5 to 15	FIRST	9/8/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 23	MW530-3	5 to 15	FIRST	9/7/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 23	MWOR-5	5 to 15	FIRST	5/17/2012	VOCs, MNA	Inactive																										
OU-2A	**IR Site 9**	**Status: Institutional Controls in-place (no GW monitoring required)**																															
OU-2A	IR Site 9	9EMW01	Unknown	UNDEFINED	5/23/2013	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	9EMW03	Unknown	UNDEFINED	5/23/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9EMW04	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW01L	Unknown	UNDEFINED	5/23/2013	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	9IF-MW01S	Unknown	UNDEFINED	5/30/2013	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	9IF-MW01U	Unknown	UNDEFINED	5/23/2013	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	9IF-MW02L	Unknown	UNDEFINED	5/23/2013	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	9IF-MW02U	Unknown	UNDEFINED	5/23/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW03L	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW03U	Unknown	UNDEFINED	5/23/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW04L	Unknown	UNDEFINED	5/30/2013	Metals, SVOCs,	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						VOCs																											
OU-2A	IR Site 9	9IF-MW04U	Unknown	UNDEFINED	5/30/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW05L	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW05U	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW06L	Unknown	UNDEFINED	5/29/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW06U	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW07L	Unknown	UNDEFINED	5/29/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW07U	Unknown	UNDEFINED	5/28/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW08L	Unknown	UNDEFINED	5/28/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW08U	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW09L	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	9IF-MW09U	Unknown	UNDEFINED	12/27/2005	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW10L	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	9IF-MW10U	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2A	IR Site 9	9IF-MW11U	Unknown	UNDEFINED	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	9IF-MW12U	Unknown	UNDEFINED	12/29/2005	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	D09-01	50 to 60	SECOND	5/28/2013	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	F9SMW01	Unknown	UNDEFINED	6/8/2006	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	F9SMW02	Unknown	UNDEFINED	6/7/2006	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	F9SMW03	Unknown	UNDEFINED	5/30/2013	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	F9SMW04	Unknown	UNDEFINED	5/28/2013	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	F9SMW05	Unknown	UNDEFINED	5/28/2013	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	F9SMW06	Unknown	UNDEFINED	6/9/2006	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	MW410-1	5 to 15	FIRST	9/29/2008	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	MW410-2	5 to 15	FIRST	5/28/2013	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	MW410-3	5 to 15	FIRST	5/28/2013	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	MW410-4	5 to 15	FIRST	9/30/2008	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	P-9-IWI-01	Unknown	FIRST	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2A	IR Site 9	P-9-IWS-01	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	P-9-MWI-01	Unknown	FIRST	9/9/2009	VOCs	Inactive																										
OU-2A	IR Site 9	P-9-MWI-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	P-9-MWI03	Unknown	FIRST	9/9/2009	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	P-9-MWI-04	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	P-9-MWI05	Unknown	FIRST	5/24/2013	Metals, SVOCs, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	P-9-MWI06	Unknown	FIRST	5/24/2013	Metals, SVOCs, VOCs	Inactive																										
OU-2A	IR Site 9	P-9-MWI-07	Unknown	FIRST	9/9/2009	VOCs	Inactive																										
OU-2A	IR Site 9	P-9-MWI-08	Unknown	FIRST	5/18/2012	VOCs	Inactive																										
OU-2A	IR Site 9	P-9-MWI-09	Unknown	FIRST	5/18/2012	VOCs	Inactive																										
OU-2A	IR Site 9	P-9-MWI-10	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	P-9-MWS-01	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	P-9-MWS-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	P-9-MWS-03	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	P-9-MWS-04	Unknown	FIRST	NA	NA	Inactive																										
OU-2A	IR Site 9	PEZ-S09-05	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 9	PEZ-S09-06	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 9	PEZ-S09-07	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 9	PEZ-S09-08	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 9	PEZ-S09-09	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2A	IR Site 9	S9-TT-MW01	6 to 16	FIRST	8/21/2013	Metals TPH, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	S9-TT-MW02	30 to 40	SECOND	8/21/2013	Metals, VOCs, MNA	Inactive																										
OU-2A	IR Site 9	S9-TT-MW03	5 to 15	FIRST	8/21/2013	Metals, VOCs, MNA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2A	IR Site 9	S9-TT-MW04	Unknown	UNDEFINED	9/1/2009	TPH, VOCs	Inactive																										
OU-2A	IR Site 9	S9-TT-MW05	Unknown	UNDEFINED	2/19/2010	VOCs	Inactive																										
OU-2B	CAA-11	37-MJ-MW-4	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	CAA-11	37-MJ-MW7	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	**IR Site 11**	**Status: VOCs in shallow groundwater, in-situ bioremediation, groundwater monitoring, and institutional controls (ROD pending, RD in progress)**																															
OU-2B	IR Site 11	37-MJ-MW1	4.2 to 12	FIRST	4/30/2012	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	37-MJ-MW3	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 11	37-MJ-MW-5	4.2 to 12	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	CA11-01	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	CA11-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	CA11-10	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	CA11-11	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	CA11-12	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	CA11-13	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	D11-01	50 to 60	SECOND	9/25/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	M03-14	27 to 36.5	FIRST	9/29/2008	VOCs	Inactive																										
OU-2B	IR Site 11	M03-15	Unknown	SECOND	2/2/2011	Metals, SVOCs, VOCs	Inactive																										
OU-2B	IR Site 11	M03-16	24.5 to 34	FIRST	4/30/2012	Metals, SVOCs TPH, VOCs	Inactive																										
OU-2B	IR Site 11	M11-01	Unknown	*Shallow*	5/16/2012	Metals TPH, VOCs, MNA	Active																										A
OU-2B	IR Site 11	M11-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	M11-03	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 11	M11-04	5 to 12	FIRST	9/25/2008	Metals TPH, VOCs,	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						MNA																											
OU-2B	IR Site 11	M11-04-OLD	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 11	M11-05	4 to 14	FIRST	9/25/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW01A	20 to 30	*Shallow*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW01B	30 to 40	*Shallow*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW01C	40 to 50	*Shallow*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW01D	50 to 60	*Deep*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW01E	58 to 68	*Deep*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW02A	Unknown	UNDEFINED	2/18/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW02B	Unknown	UNDEFINED	2/22/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW02C	Unknown	UNDEFINED	2/22/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW02D	Unknown	UNDEFINED	2/22/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW02E	Unknown	UNDEFINED	2/22/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW03A	20 to 30	*Shallow*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW03B	30 to 40	*Shallow*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW03C	40 to 50	*Shallow*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW03D	50 to 60	*Deep*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW03E	60 to 70	*Deep*	5/19/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW04A	25 to 30	*Shallow*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q		Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW04B	30 to 40	*Shallow*	5/20/2014	VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW04C	40 to 50	*Shallow*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW04D	50 to 60	*Deep*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW04E	60 to 70	*Deep*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW05A	20 to 30	*Shallow*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW05B	30 to 40	*Shallow*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW05C	39.5 to 49.5	*Shallow*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2B	IR Site 11	S11-TT-MW05D	49.5 to 59.5	*Deep*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW05E	60 to 70	*Deep*	5/20/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 11	S11-TT-MW06A	Unknown	UNDEFINED	2/15/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW06B	Unknown	UNDEFINED	2/14/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW06C	Unknown	UNDEFINED	2/14/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW06D	Unknown	UNDEFINED	2/14/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 11	S11-TT-MW06E	Unknown	UNDEFINED	2/25/2011	Metals, VOCs, MNA	Inactive																										
OU-2B	**IR Site 21**	**Status: VOCs in shallow groundwater, in-situ bioremediation, groundwater monitoring, and institutional controls (ROD pending, RD in progress)**																															
OU-2B	IR Site 21	398-A19	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 21	398-MW1	2.41 to 12.41	*Shallow*	3/3/2014	Metals TPH, VOCs	Active	Q																									Q
OU-2B	IR Site 21	398-MW2	2.5 to 12.5	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 21	CA03-01	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 21	M03-17	24 to 33.5	FIRST	9/19/2005	VOCs	Inactive																										
OU-2B	IR Site 21	M07B-01	4 to 11	*Shallow*	5/21/2013	Metals TPH, VOCs, MNA	Active																										A
OU-2B	IR Site 21	M11-06	4 to 14	*Shallow*	5/22/2014	Metals TPH, VOCs, MNA	Active	S																									S
OU-2B	IR Site 21	OU2B-MW01	7 to 12	*Shallow*	12/2/2013	Metals TPH, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	OU2B-MW02	7 to 12	*Shallow*	12/2/2013	Metals TPH, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	OU2B-MW03	7 to 12	*Shallow*	12/3/2013	Metals TPH, VOCs, MNA	Active																										S
OU-2B	IR Site 21	OU2B-MW04	7 to 12	*Shallow*	12/3/2013	Metals TPH, VOCs, MNA	Active																										S
OU-2B	IR Site 21	OU2B-MW05	7 to 12	*Shallow*	12/3/2013	Metals TPH, VOCs, MNA	Active																										S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2B	IR Site 21	S21-TT-MW01A	20 to 30	*Shallow*	8/31/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW01B	30 to 40	*Shallow*	8/31/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW01C	40 to 50	*Shallow*	8/31/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW01D	50 to 60	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 21	S21-TT-MW01E	60 to 70	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 21	S21-TT-MW02A	20 to 30	*Shallow*	8/28/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW02B	30 to 40	*Shallow*	8/30/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW02C	40 to 50	*Shallow*	8/28/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW02D	50 to 60	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 21	S21-TT-MW02E	59 to 69	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 21	S21-TT-MW03A	25 to 30	*Shallow*	8/30/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW03B	30 to 40	*Shallow*	8/30/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW03C	40 to 50	*Shallow*	8/30/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW03D	50 to 60	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 21	S21-TT-MW03E	60 to 70	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 21	S21-TT-MW05A	20 to 30	*Shallow*	8/27/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW05B	30 to 40	*Shallow*	8/27/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW05C	40 to 50	*Shallow*	8/27/2012	Metals, VOCs, MNA	Active																										Q
OU-2B	IR Site 21	S21-TT-MW05D	50 to 60	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q		Q	Q													Q
OU-2B	IR Site 21	S21-TT-MW05E	59 to 69	*Deep*	5/21/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 21	WA-8	5 to 15	FIRST	9/14/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	**IR Site 3**	**Status: Institutional controls and groundwater monitoring for OU-2B plume (ROD pending, RD in progress)**																															
OU-2B	IR Site 3	398-MW3	2.9 to 12.9	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 3	398-MW4	2.9 to 12.9	FIRST	5/15/2012	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 3	D03-01	49.5 to 59.5	SECOND	9/24/2008	Metals TPH, VOCs,	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						MNA																											
OU-2B	IR Site 3	M03-04	3 to 11	FIRST	9/24/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 3	M03-07	3 to 13	FIRST	5/18/2012	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 3	M03-08A	3 to 11	FIRST	2/12/2010	Metals, SVOCs TPH, VOCs	Inactive																										
OU-2B	IR Site 3	M03-09	21 to 33	FIRST	9/24/2008	Metals TPH, VOCs	Inactive																										
OU-2B	IR Site 3	MW97-1	28 to 38	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 3	MW97-2	26 to 36	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 3	MW97-3	5 to 15	FIRST	5/22/2012	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 3	S3-TT-MW01	5 to 10	*Shallow*	5/30/2014	Metals, VOCs, MNA	Active					Q[4]																					Q
OU-2B	IR Site 3	S3-TT-MW02	Unknown	UNDEFINED	9/10/2009	Metals TPH	Inactive																										
OU-2B	**IR Site 4**	**Status: VOCs in shallow groundwater, in-situ bioremediation, groundwater monitoring, and institutional controls (ROD pending, RD in progress)**																															
OU-2B	IR Site 4	372-MW1	2.5 to 12.5	*Shallow*	5/23/2014	Metals TPH, VOCs, MNA	Active	Q							Q	Q	Q		Q	Q													Q
OU-2B	IR Site 4	372-MW3	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 4	CA04-01	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 4	CA04-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 4	D03-02	46.5 to 56.5	*Deep*	5/22/2014	Metals, VOCs	Active	S																									S
OU-2B	IR Site 4	D03-03	48 to 58	*Deep*	5/30/2014	Metals, VOCs, MNA	Active	Q					Q		Q	Q	Q		Q	Q													Q
OU-2B	IR Site 4	D03-04	47 to 57	*Deep*	5/23/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q		Q	Q													Q
OU-2B	IR Site 4	D04-01	86 to 96	SECOND	3/18/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 4	D04-02	86.5 to 96.5	SECOND	3/18/2008	Metals TPH, VOCs, MNA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2B	IR Site 4	D04-03	84 to 94	SECOND	9/25/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 4	IW-01	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	M03-05	3 to 13	FIRST	9/24/2008	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 4	M03-06	3 to 12	*Shallow*	5/23/2014	Metals TPH, VOCs, MNA	Active	S																									S
OU-2B	IR Site 4	M03-10	21 to 33	*Shallow*	5/23/2014	Metals, SVOCs TPH, VOCs, MNA	Active	Q							Q	Q	Q		Q	Q													Q
OU-2B	IR Site 4	M03-11	26 to 36	*Shallow*	5/23/2014	Metals TPH, VOCs	Active	S																									S
OU-2B	IR Site 4	M03-12	26 to 36	FIRST	9/25/2008	Metals TPH, VOCs	Inactive																										
OU-2B	IR Site 4	M03-13	26.5 to 36.5	*Shallow*	6/2/2014	Metals TPH, VOCs	Active	S																									S
OU-2B	IR Site 4	M04-05	Unknown	*Shallow*	5/23/2014	Metals TPH, VOCs, MNA	Active						Q																				Q
OU-2B	IR Site 4	M04-06	Unknown	*Shallow*	6/2/2014	Metals TPH, VOCs, MNA	Active	S																									S
OU-2B	IR Site 4	M04-07	Unknown	FIRST	NA	NA	Inactive																										
OU-2B	IR Site 4	MBG-3	5 to 15	FIRST	9/15/2005	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	IR Site 4	MW-01	Unknown	SECOND	6/6/2008	VOCs	Inactive																										
OU-2B	IR Site 4	MW-02	Unknown	SECOND	6/6/2008	VOCs	Inactive																										
OU-2B	IR Site 4	MW-03	Unknown	SECOND	6/6/2008	VOCs	Inactive																										
OU-2B	IR Site 4	MW-04	Unknown	SECOND	6/6/2008	VOCs	Inactive																										
OU-2B	IR Site 4	MW360-1	5 to 15	*Shallow*	5/22/2014	Metals, SVOCs TPH, VOCs, MNA	Active	A																									A
OU-2B	IR Site 4	MW360-2	Unknown	*Shallow*	5/22/2014	Cyanide, Metals, SVOCs TPH, VOCs,	Active	S																									S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						MNA																											
OU-2B	IR Site 4	MW360-3	5 to 15	*Shallow*	5/22/2014	Metals TPH, VOCs, MNA	Active	S																									S
OU-2B	IR Site 4	MW360-4	5 to 15	*Shallow*	5/22/2014	Metals, SVOCs TPH, VOCs, MNA	Active	A																									A
OU-2B	IR Site 4	MW4-2-1	20 to 25	*Shallow*	5/23/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	MW4-2-11	5 to 10	*Shallow*	6/2/2014	Cyanide, Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	MW4-2-12	26 to 31	*Shallow*	6/2/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	MW4-2-4	25.5 to 30.5	*Shallow*	5/23/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	MW4-2-6	15 to 20	*Shallow*	6/2/2014	Cyanide, Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	P-11-MWI-03	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	P-11-MWI-07	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	P-11-MWI-11	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWD1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWD2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWD3	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWD4	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWD5	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWD6	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWIA1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWIA2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWIA3	Unknown	UNDEFINED	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2B	IR Site 4	P-4-1-MWIB1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWIB2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWIB3	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWS1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWS2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWS3	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWS4	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWS5	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	P-4-1-MWS6	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41IW01	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41IW02	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41IW03	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41IW04	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41IW05	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41IW06	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW01	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW02	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW03	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW04	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW05	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW06	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW07	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	PP41MW08	Unknown	SECOND	NA	NA	Inactive																										
OU-2B	IR Site 4	S4-TT-MW01	3 to 8	*Shallow*	5/22/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW02A	22 to 32	*Shallow*	5/22/2014	Metals, SVOCs, VOCs, MNA	Active	Q							Q	Q	Q	Q	Q	Q													Q

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2B	IR Site 4	S4-TT-MW02B	30 to 40	*Shallow*	5/22/2014	Metals, SVOCs, VOCs, MNA	Active	Q							Q	Q	Q		Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW02C	40 to 50	*Shallow*	5/22/2014	Metals, VOCs, MNA	Active	Q							Q	Q	Q		Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW02D	50 to 60	*Deep*	5/22/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW02E	60 to 70	*Deep*	5/22/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW03A	22 to 32	*Shallow*	5/23/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW03B	30 to 40	*Shallow*	5/23/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW03C	40 to 50	*Deep*	5/23/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW03D	50 to 60	*Deep*	5/23/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	S4-TT-MW03E	60 to 70	*Deep*	5/23/2014	Metals, SVOCs, VOCs, MNA	Active	Q					Q		Q	Q	Q	Q	Q	Q													Q
OU-2B	IR Site 4	ZVI-MW07A2	Unknown	UNDEFINED	2/17/2010	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 4	ZVI-MW07A3	Unknown	UNDEFINED	2/18/2010	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 4	ZVI-MW07B	Unknown	UNDEFINED	2/18/2010	Metals, VOCs, MNA	Inactive																										
OU-2B	IR Site 4	ZVI-MW07C	Unknown	UNDEFINED	2/18/2010	Metals, VOCs, MNA	Inactive																										
OU-2B	N/A	CA11-09	Unknown	FIRST	NA	NA	Inactive																										
OU-2C	**IR Site 10**	**Status: Remedial Design in progress, not currently part of BGMP (ROD in place)**																															
OU-2C	IR Site 10	CA05-05	Unknown	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 10	D10A-01	Unknown	SECOND	NA	NA	Inactive																										
OU-2C	IR Site 10	M10-01	4 to 14	FIRST	6/4/2012	Metals, PAHs, Pesticides, SVOCs	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						TPH, VOCs, MNA																											
OU-2C	IR Site 10	M10-02	3 to 13	FIRST	6/4/2012	Metals, PAHs, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2C	IR Site 10	M10-03	3 to 13	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 10	PEZ-S10A-02	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 10	PEZ-S10A-05	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	**IR Site 12**	**Status: Remedial Design in progress, not currently part of BGMP (ROD in place)**																															
OU-2C	IR Site 12	D12-01	60 to 70	SECOND	3/25/2008	Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2C	IR Site 12	M12-01	5 to 15	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 12	M12-02	5 to 15	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 12	M12-03	4 to 14	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 12	M12-04	4 to 14	FIRST	3/27/2007	Metals TPH, VOCs, MNA	Inactive																										
OU-2B	**IR Site 5**	**Status: Anticipated remedial action to start in late 2015 ISCO, enhanced bioremediation, groundwater monitoring, and institutional controls (ROD in place)**																															
OU-2C	IR Site 5	1MW6S	9.5 to 14.5	FWBZ	6/2/2014	Metals, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	1MW7S	Unknown	UNDEFINED	3/16/2005	VOCs	Inactive																										
OU-2C	IR Site 5	282-MW1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	282-MW2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	282-MW3	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	2MW8S	8.5 to 13.5	FWBZ	6/2/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, MNA	Active	S	S				S		A	A	A	A	A														S
OU-2C	IR Site 5	3MW10S	Unknown	UNDEFINED	5/27/2009	VOCs	Inactive																										
OU-2C	IR Site 5	400MJ-MW1	Unknown	UNDEFINED	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2C	IR Site 5	400MJ-MW2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	400MJ-MW3	Unknown	UNDEFINED	4/30/2007	Cyanide, Metals, SVOCs, VOCs	Inactive																										
OU-2C	IR Site 5	5-3MW1S	9 to 12	FWBZ	5/30/2014	Metals, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	5-3MW6S	10 to 13	FWBZ	5/30/2014	Metals, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	5-3MW7S	9 to 12	FWBZ	5/30/2014	Metals, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	CA05-02	Unknown	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	CA05-03	Unknown	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	CA05-04	Unknown	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	D05-01	60 to 70	SECOND	3/25/2008	Cyanide, Metals, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2C	IR Site 5	D05-02	Unknown	SECOND	NA	NA	Inactive																										
OU-2C	IR Site 5	D05-03	57 to 67	SWBZ	5/27/2014	Cyanide, Metals, SVOCs TPH, VOCs	Active	A																									A
OU-2C	IR Site 5	D05-04	65 to 70	SWBZ	5/30/2014	Cyanide, Metals, SVOCs, VOCs, MNA	Active	S	A							A	A	A	A														S
OU-2C	IR Site 5	D05-05	65 to 70	SWBZ	5/23/2014	Cyanide, Metals, SVOCs, VOCs, MNA	Active	A	A							A	A	A	A														A
OU-2C	IR Site 5	D05-06	65.6 to 70.6	SWBZ	5/23/2014	SVOCs, VOCs, MNA	Active	A																									A
OU-2C	IR Site 5	D05-07	65 to 70	SECOND	8/29/2005	VOCs	Inactive																										
OU-2C	IR Site 5	D05-08	65 to 70	SECOND	8/30/2005	VOCs	Inactive																										
OU-2C	IR Site 5	D05-09	60 to 70	SWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs,	Active	A	A							A	A	A	A														A

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						SVOCs TPH, VOCs, MNA																											
OU-2C	IR Site 5	L05-01	20 to 30	FIRST	2/19/2009	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2C	IR Site 5	L05-02	20 to 30	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	A	A						A	A	A	A	A	A													A
OU-2C	IR Site 5	L05-03	15 to 25	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	A	A						A	A	A	A	A	A													A
OU-2C	IR Site 5	L05-04	20 to 30	FWBZ	5/30/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S				S	S	S	S	S	S	S	S													S
OU-2C	IR Site 5	L05-05	20 to 30	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	L05-06	20 to 30	FWBZ	5/23/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-01	4 to 14	FIRST	5/1/2007	Cyanide, Metals, SVOCs TPH, VOCs,	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						MNA																											
OU-2C	IR Site 5	M05-02	4 to 14	FWBZ	5/27/2014	Cyanide, Metals, PAHs, SVOCs, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-03	4 to 12	FIRST	5/4/2007	Cyanide, Metals, SVOCs TPH, VOCs	Inactive																										
OU-2C	IR Site 5	M05-04	5 to 15	FWBZ	5/27/2014	Metals, PAHs, SVOCs TPH, VOCs, MNA	Active	S	S				S		A	A	A	A	A														S
OU-2C	IR Site 5	M05-05	4 to 14	FWBZ	5/23/2014	Cyanide, Metals, PAHs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-06	3 to 13	FWBZ	5/27/2014	Cyanide, Metals, PAHs, SVOCs TPH, VOCs	Active	S	S				S																				S
OU-2C	IR Site 5	M05-07	Unknown	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	M05-08	7 to 15	FWBZ	5/23/2014	Metals, PAHs, SVOCs TPH, VOCs, MNA	Active	A	A				A		A	A	A	A	A														A
OU-2C	IR Site 5	M05-09	5 to 15	FWBZ	5/23/2014	Cyanide, Metals, PAHs, SVOCs TPH, VOCs, MNA	Active	A	A				A		A	A	A	A	A														A
OU-2C	IR Site 5	M05-10	Unknown	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	M05-11	5 to 15	FWBZ	5/30/2014	Metals, PAHs, SVOCs TPH, VOCs, MNA	Active	S	S				S		A	A	A	A	A														S
OU-2C	IR Site 5	M05-12	5.5 to 15.5	FIRST	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2C	IR Site 5	M05-13	10 to 15	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-14	10 to 15	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	A	A						A	A	A	A	A	A													A
OU-2C	IR Site 5	M05-15	10 to 15	FWBZ	5/30/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S				S	S	S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-16	10 to 15	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-17	5.5 to 15.5	FWBZ	5/30/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-18	4 to 14	FWBZ	6/2/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	M05-19	10 to 15	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2C	IR Site 5	M05-20	10 to 15	FWBZ	5/27/2014	Cyanide, Metals, PAHs, PCBs, SVOCs TPH, VOCs, MNA	Active	S	S				S	S	S	S	S	S	S	S													S
OU-2C	IR Site 5	M05BS-01	5 to 15	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	M05HW-01	5 to 15	FWBZ	6/2/2014	Metals, PAHs, SVOCs TPH, VOCs, MNA	Active	S	S				S		A	A	A	A	A														S
OU-2C	IR Site 5	M05-P	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	M08-07	Unknown	UNDEFINED	3/28/2007	Metals, Pesticides, SVOCs TPH, VOCs, MNA	Inactive																										
OU-2C	IR Site 5	M110-A	3.5 to 13.5	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	M111-A	3.5 to 13.5	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	M115-E	7 to 12	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	M116-E	10 to 20	FIRST	NA	NA	Inactive																										
OU-2C	IR Site 5	M117-E	Unknown	UNDEFINED	2/24/2009	Metals TPH, VOCs, MNA	Inactive																										
OU-2C	IR Site 5	MLS-1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	MLS-7	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	MRW-1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	MRW-2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	MRW-3	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	MRW-4	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	MW 2A-AE	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	P-5-1-MWD1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	P-5-1-MWD2	Unknown	UNDEFINED	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-2C	IR Site 5	P-5-1-MWD3	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	P-5-1-MWD4	Unknown	UNDEFINED	5/2/2007	Cyanide, Metals, SVOCs, VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWD5	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	P-5-1-MWI1	Unknown	UNDEFINED	3/14/2005	VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWI2	Unknown	UNDEFINED	3/15/2005	VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWI3	19 to 22	FWBZ	5/23/2014	Metals, SVOCs TPH, VOCs, MNA	Active	S	S						S	S	S	S	S	S													S
OU-2C	IR Site 5	P-5-1-MWI4	Unknown	UNDEFINED	5/1/2007	Cyanide, Metals, SVOCs, VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWI5	Unknown	UNDEFINED	3/16/2005	VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWS1	Unknown	UNDEFINED	3/14/2005	VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWS2	Unknown	UNDEFINED	3/14/2005	VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWS3	Unknown	UNDEFINED	3/15/2005	VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWS4	Unknown	UNDEFINED	5/2/2007	Cyanide, Metals, SVOCs, VOCs	Inactive																										
OU-2C	IR Site 5	P-5-1-MWS5	Unknown	UNDEFINED	3/15/2005	VOCs	Inactive																										
OU-2C	IR Site 5	PEZ-S05-09	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-2C	IR Site 5	PEZ-S05-10	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-3	**IR Site 1**	**Status: Remedial action in progress, ISCO and institutional controls. Anticipated completion late 2015. LTM in BGMP thereafter (ROD in place)**																															
OU-3	IR Site 1	M001-A	4 to 14	FIRST	5/30/2012	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, RAD	Inactive																										
OU-3	IR Site 1	M001-B	62 to 72	SECOND	5/29/2012	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										
OU-3	IR Site 1	M001-E	9.5 to 19.5	FWBZ	12/11/2013	Cyanide, Metals, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-3	IR Site 1	M002-A	4 to 10	FWBZ	12/11/2013	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
OU-3	IR Site 1	M002-E	10 to 20	FIRST	NA	NA	Inactive																										
OU-3	IR Site 1	M003-A	4 to 14	FWBZ	12/10/2013	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
OU-3	IR Site 1	M003-B	33 to 43	SECOND	5/30/2012	Cyanide, Metals, SVOCs, VOCs, MNA, RAD	Inactive																										
OU-3	IR Site 1	M003-E	11.25 to 21.25	FIRST	NA	NA	Inactive																										
OU-3	IR Site 1	M004-A	3.5 to 13.5	FIRST	5/30/2012	Cyanide, Metals, SVOCs, VOCs, MNA, RAD	Inactive																										
OU-3	IR Site 1	M01-P	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-3	IR Site 1	M026-A	3.5 to 13.5	FWBZ	12/11/2013	Cyanide, Metals, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
OU-3	IR Site 1	M026-E	Unknown	FIRST	8/26/2005	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										
OU-3	IR Site 1	M027-A	3.5 to 13.5	FWBZ	12/11/2013	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, MNA, RAD	Active																			S	S	S	S	S	S	S	S
OU-3	IR Site 1	M027-B	56 to 66	SECOND	5/31/2012	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										
OU-3	IR Site 1	M027-C	80 to 90	SECOND	5/30/2012	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-3	IR Site 1	M027-E	12 to 22	FIRST	8/26/2005	VOCs	Inactive																										
OU-3	IR Site 1	M028-A	4 to 14	FWBZ	12/11/2013	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, MNA, RAD	Active	S				A		A	A	A	A	A	A	A						S	S	S	S	S	S	S	S
OU-3	IR Site 1	M028-C	80 to 90	SECOND	5/30/2012	Cyanide, Metals, SVOCs, VOCs, MNA, RAD	Inactive																										
OU-3	IR Site 1	M028-E	11 to 21	FWBZ	12/11/2013	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, MNA, RAD	Active	S				A		A	A	A	A	A	A	A						S	S	S	S	S	S	S	S
OU-3	IR Site 1	M029-A	4 to 14	FWBZ	12/11/2013	Cyanide, Metals, SVOCs, VOCs, RAD	Active	S																		S	S	S	S	S	S	S	S
OU-3	IR Site 1	M029-E	10 to 20	FIRST	8/26/2005	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										
OU-3	IR Site 1	M030-A	2.5 to 12.5	FWBZ	12/11/2013	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, RAD	Active	S																		S	S	S	S	S	S	S	S
OU-3	IR Site 1	M030-C	76.5 to 86.5	SECOND	5/29/2012	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										
OU-3	IR Site 1	M030-E	6 to 16	FIRST	NA	NA	Inactive																										
OU-3	IR Site 1	M031-C	77.5 to 87.5	SECOND	5/30/2012	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										
OU-3	IR Site 1	M031-E	8 to 18	FIRST	8/25/2005	VOCs	Inactive																										
OU-3	IR Site 1	M033-A	4.25 to 12.5	FWBZ	12/12/2013	Cyanide, Dioxins, Metals, PCBs, Pesticides, SVOCs,	Active	S																		S	S	S	S	S	S	S	S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						VOCs, MNA, RAD																											
OU-3	IR Site 1	M034-A	4.25 to 12.5	FWBZ	12/11/2013	Cyanide, Metals, SVOCs, VOCs, MNA, RAD	Active	S							A		A	A	A	A	A	A	A			S	S	S	S	S	S	S	S
OU-3	IR Site 1	M035-A	4 to 14	FWBZ	12/12/2013	Cyanide, Metals, SVOCs, VOCs, MNA, RAD	Active																			S	S	S	S	S	S	S	S
OU-3	IR Site 1	M2A	Unknown	SECOND	NA	NA	Inactive																										
OU-3	IR Site 1	MG1-01-S	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	**IR Site 2**	**Status: MNA, engineering controls, and institutional controls (Included in BGMP until Parcel transfer to VA)**																															
OU-4A	IR Site 2	442-MW1	3.5 to 13.5	FIRST	12/10/2012	Cyanide, Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	442-MW2	Unknown	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	442-MW3	Unknown	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	CA01-01	Unknown	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	DRA-01	35 to 45	FIRST	6/5/2013	RAD	Inactive																										
OU-4A	IR Site 2	GP-01	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	GP-02	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	GP-03	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	GP-04	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	GP-05	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	GP-06	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	GP-07	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	GP-08	Unknown	VADOSE	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-4A	IR Site 2	GP-09	Unknown	VADOSE	NA	NA	Inactive																										
OU-4A	IR Site 2	M009-A	3.5 to 13.5	FIRST	8/28/2012	VOCs, MNA	Inactive																										
OU-4A	IR Site 2	M010-A	5.5 to 13.5	FIRST	5/30/2012	VOCs	Inactive																										
OU-4A	IR Site 2	M010-B	35.5 to 43.25	SECOND	12/6/2012	Metals, PAHs, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	M011-A	3.5 to 13.5	FIRST	12/6/2012	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M012-A	5 to 15	FIRST	12/6/2012	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	M012-B	63.5 to 73.5	SECOND	12/11/2012	Metals, PAHs, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	M013-A	3.5 to 13.5	FIRST	12/6/2012	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M013-C	69 to 79	SECOND	12/6/2012	Metals, PAHs, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M014-A	4.5 to 14.5	FIRST	12/6/2012	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-4A	IR Site 2	M014-B	49.5 to 59.5	SECOND	12/6/2012	Metals, PAHs, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M015-A	4.5 to 14.5	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	M016-A	4 to 14	FWBZ	6/3/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M016-B	45.5 to 55.5	SECOND	12/10/2012	Metals, PAHs, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	M016-E	14 to 24	FWBZ	6/3/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M017-A	4 to 14	FIRST	12/10/2012	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M018-A	4 to 14	FWBZ	6/3/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M018-E	36 to 46	FWBZ	6/3/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M019-A	5 to 15	FIRST	5/23/2012	Metals, PAHs, PCBs, Pesticides,	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						SVOCs TPH, VOCs, RAD																											
OU-4A	IR Site 2	M019-E	30.5 to 40.5	FIRST	12/11/2012	Metals, PAHs, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M020-A	3.5 to 13.5	FWBZ	6/3/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M020-B	55 to 65	SECOND	12/11/2012	Metals, PAHs, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M020-E	27.5 to 34.5	FWBZ	6/4/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M021-A	3.5 to 13.5	FWBZ	6/4/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M021-C	82 to 92	SECOND	12/11/2012	Metals, PAHs, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M021-E	30 to 37	FWBZ	6/4/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M022-A	3.5 to 13.5	FWBZ	6/4/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs,	Active				S	S[5]																					S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						RAD																											
OU-4A	IR Site 2	M022-E	27.25 to 37.5	FWBZ	6/5/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M023-A	3.5 to 13.5	FWBZ	6/4/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M023-B	83 to 93	SECOND	12/10/2012	Metals, PAHs, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M023-C	99.5 to 109.5	SECOND	12/10/2012	Metals, PAHs, SVOCs TPH, VOCs, RAD	Inactive																										
OU-4A	IR Site 2	M023-E	25 to 35	FWBZ	6/4/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, RAD	Active				S	S[5]																					S
OU-4A	IR Site 2	M024-A	4 to 14	FWBZ	6/4/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Active	S		S	S	S[5]																					S
OU-4A	IR Site 2	M024-E	22 to 32	FWBZ	6/4/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, RAD	Active	S		S	S	S[5]																					S
OU-4A	IR Site 2	M036-A	5 to 15	FWBZ	6/5/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs,	Active	S		S	S	S[5]																					S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						MNA, RAD																											
OU-4A	IR Site 2	M036-B	90 to 100	SECOND	12/10/2012	Metals, PAHs, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	M036-E	20 to 30	FWBZ	6/5/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active	S		S	S	S[5]																					S
OU-4A	IR Site 2	M037-A	8 to 18	FWBZ	6/5/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active	S		S	S	S[5]																					S
OU-4A	IR Site 2	M037-B	77 to 87	SECOND	12/11/2012	Metals, PAHs, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	M037-E	15.5 to 25.5	FWBZ	6/5/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active	S		S	S	S[5]																					S
OU-4A	IR Site 2	M038-A	5 to 15	FWBZ	6/13/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active	S		S	S	S[5]																					S
OU-4A	IR Site 2	M038-B	70 to 80	SECOND	12/11/2012	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-4A	IR Site 2	M038-E	15 to 25	FWBZ	6/5/2014	Metals, PAHs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active	S		S	S	S[5]																					
OU-4A	IR Site 2	M039-A	5 to 15	FWBZ	6/13/2014	Metals, PAHs, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Active	S		S	S	S[5]																					
OU-4A	IR Site 2	M039-B	52 to 62	SECOND	12/11/2012	Metals, PAHs, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
OU-4A	IR Site 2	M039-E	Unknown	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	M040-A	Unknown	FWBZ	6/13/2014	Metals, Pesticides	Active				Q	Q[5]																					Q
OU-4A	IR Site 2	M040-E	Unknown	FWBZ	6/13/2014	Metals, Pesticides	Active				Q	Q[5]																					Q
OU-4A	IR Site 2	M041-A	Unknown	FWBZ	6/13/2014	Metals, Pesticides	Active				Q	Q[5]																					Q
OU-4A	IR Site 2	M041-E	Unknown	FWBZ	6/13/2014	Metals, Pesticides	Active				Q	Q[5]																					Q
OU-4A	IR Site 2	M042-A	Unknown	FWBZ	6/13/2014	Metals, Pesticides, SVOCs, VOCs	Active	Q		Q	Q	Q[5]																					Q
OU-4A	IR Site 2	M042-E	Unknown	FWBZ	6/13/2014	Metals, Pesticides, SVOCs, VOCs	Active	Q		Q	Q	Q[5]																					Q
OU-4A	IR Site 2	M043-A	Unknown	FWBZ	6/13/2014	Metals, Pesticides, SVOCs	Active			Q	Q	Q[5]																					Q
OU-4A	IR Site 2	M043-E	Unknown	FWBZ	6/13/2014	Metals, Pesticides, SVOCs	Active			Q	Q	Q[5]																					Q
OU-4A	IR Site 2	M103-A	5.5 to 15.5	FIRST	8/28/2012	VOCs, MNA	Inactive																										
OU-4A	IR Site 2	M103-B	31.5 to 37.5	SECOND	8/28/2012	VOCs, MNA	Inactive																										
OU-4A	IR Site 2	M104-A	3.5 to 13.5	FIRST	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-4A	IR Site 2	M105-A	5.5 to 15.5	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	M105-B	58.5 to 68.5	SECOND	NA	NA	Inactive																										
OU-4A	IR Site 2	M106-A	3.5 to 13.5	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	M107-A	3.5 to 13.5	FIRST	NA	NA	Inactive																										
OU-4A	IR Site 2	M108-A	6 to 16	FIRST	12/6/2012	Metals, PAHs, VOCs, MNA	Inactive																										
OU-4A	IR Site 2	M108-B	47.5 to 57.5	SECOND	12/6/2012	RAD	Inactive																										
OU-4A	IR Site 2	M109-A	6 to 16	FIRST	NA	NA	Inactive																										
OU-5	**IR Site 25**	**Status: NFA proposed, ROD amendment forthcoming (ROD Amendment NFA)**																															
OU-5	IR Site 25	BZMW3	Unknown	FIRST	2/29/2012	VOCs, MNA	Inactive																										
OU-5	IR Site 25	BZMW5	Unknown	FIRST	2/29/2012	VOCs, MNA	Inactive																										
OU-5	IR Site 25	D-02R	Unknown	UNDEFINED	4/3/2007	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	M25-02	Unknown	FIRST	6/16/2008	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	M25-04	Unknown	FIRST	6/16/2008	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	M25-08	Unknown	FIRST	6/16/2008	PAHs, VOCs	Inactive																										
OU-5	IR Site 25	OU5-SG-17D	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	OU5-SG-17S	Unknown	VADOSE	3/15/2005	labeled as "Soil gas probe/Well", do methods available	Inactive																										
OU-5	IR Site 25	OU5-SG-18D	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	OU5-SG-18S	Unknown	VADOSE	3/15/2005	labeled as "Soil gas probe/Well", do methods available	Inactive																										
OU-5	IR Site 25	OU5-SG-19D	Unknown	VADOSE	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-5	IR Site 25	OU5-SG-19S	Unknown	VADOSE	3/15/2005	labeled as "Soil gas probe/Well", do methods available	Inactive																										
OU-5	IR Site 25	P181-MW45	Unknown	FIRST	6/17/2008	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	P181-MW46	Unknown	FIRST	8/15/2011	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	P181-MW47	Unknown	FIRST	6/17/2008	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	PMW1	Unknown	FIRST	6/16/2008	VOCs	Inactive																										
OU-5	IR Site 25	PMW2	Unknown	FIRST	8/15/2011	VOCs, MNA	Inactive																										
OU-5	IR Site 25	PMW5	Unknown	FIRST	8/15/2011	VOCs, MNA	Inactive																										
OU-5	IR Site 25	S-35	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 25	S-35R	Unknown	FIRST	6/16/2008	VOCs	Inactive																										
OU-5	IR Site 25	S-35R-2	Unknown	UNDEFINED	8/15/2011	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA1-BSW	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA1-PZ1	Unknown	FIRST	3/12/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA1-PZ2	Unknown	FIRST	3/12/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA1-PZ3	Unknown	FIRST	3/13/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA1-PZ4	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA1-PZ5	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA1-PZ6	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA1-PZ7	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA1-PZ8	Unknown	FIRST	3/12/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA1-SVE1	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	TA1-SVE2	Unknown	VADOSE	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-5	IR Site 25	TA1-SVE3	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	TA1-SVE4	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	TA2-BSW	Unknown	FIRST	3/13/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA2-PZ1	Unknown	FIRST	3/12/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA2-PZ2	Unknown	FIRST	3/12/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA2-PZ3	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA2-PZ4	Unknown	FIRST	3/12/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA2-PZ5	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA2-PZ6	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA2-PZ7	Unknown	FIRST	3/12/2008	VOCs	Inactive																										
OU-5	IR Site 25	TA2-PZ8	Unknown	FIRST	3/13/2008	VOCs, MNA	Inactive																										
OU-5	IR Site 25	TA2-SVE1	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	TA2-SVE2	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	TA2-SVE3	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 25	TA2-SVE4	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	**IR Site 30**	**Status: NFA proposed, ROD amendment forthcoming (Soil remediation complete and closed, GW part of OU-5)**																															
OU-5	IR Site 30	BZMW1	Unknown	FIRST	2/29/2012	VOCs, MNA	Inactive																										
OU-5	IR Site 30	BZMW2	Unknown	FIRST	2/29/2012	VOCs, MNA	Inactive																										
OU-5	**IR Site 31**	**Status: NFA proposed, ROD amendment forthcoming (Soil RC/SC, GW part of OU-5)**																															
OU-5	IR Site 31	BZMW4	Unknown	FIRST	2/29/2012	VOCs, MNA	Inactive																										
OU-5	IR Site 31	BZMW6	Unknown	FIRST	2/29/2012	VOCs, MNA	Inactive																										
OU-5	IR Site 31	BZMW7	Unknown	FIRST	2/29/2012	VOCs, MNA	Inactive																										
OU-5	IR Site 31	M25-01	Unknown	FIRST	2/29/2012	Metals, PAHs, PCBs, Pesticides, SVOCs, VOCs, MNA	Inactive																										
OU-5	IR Site 31	M25-06	Unknown	FIRST	8/15/2011	Metals, PAHs, PCBs, Pesticides,	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						SVOCs, VOCs, MNA																											
OU-5	IR Site 31	M25-07	Unknown	FIRST	8/15/2011	Metals, PAHs, PCBs, Pesticides, SVOCs, VOCs, MNA	Inactive																										
OU-5	IR Site 31	M25-09	Unknown	FIRST	8/15/2011	Metals, PAHs, PCBs, Pesticides, SVOCs, VOCs, MNA	Inactive																										
OU-5	IR Site 31	OU5-SG-16D	Unknown	VADOSE	NA	NA	Inactive																										
OU-5	IR Site 31	OU5-SG-16S	Unknown	VADOSE	3/15/2005	labeled as "Soil gas probe/Well", do methods available	Inactive																										
OU-5	IR Site 31	PMW4	Unknown	FIRST	6/17/2008	VOCs	Inactive																										
OU-5	IR Site 31	PMW6	Unknown	FIRST	6/16/2008	VOCs	Inactive																										
OU-5	IR Site 31	PW-10A	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 31	PW-12	Unknown	FIRST	6/16/2008	Metals, PAHs, PCBs, Pesticides, SVOCs, VOCs, MNA	Inactive																										
OU-6	**IR Site 26**	**Status: Remedial action complete, ISCO, in-situ bioremediation, groundwater monitoring, short-term institutional controls (OPS, MNA for VOCs, nearing RGs)**																															
OU-6	IR Site 26	26MW01	Unknown	UNDEFINED	6/4/2012	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 26	26MW02	Unknown	UNDEFINED	6/4/2012	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 26	26MW03	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active	Q								Q	Q	Q	Q	Q	Q	Q	Q	Q	Q								Q
OU-6	IR Site 26	26MW04	Unknown	UNDEFINED	6/4/2012	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 26	26MW05	Unknown	UNDEFINED	6/4/2012	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 26	26MW06	Unknown	UNDEFINED	1/28/2008	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 26	26MW07	Unknown	UNDEFINED	6/4/2012	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 26	26MW08	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active	Q								Q	Q	Q	Q	Q	Q	Q	Q	Q	Q								Q
OU-6	IR Site 26	26MW09	Unknown	UNDEFINED	6/4/2012	Metals, VOCs, MNA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-6	IR Site 26	26PZ01	Unknown	FWBZ	6/2/2014	Metals, VOCs, MNA	Active	Q								Q	Q	Q	Q	Q	Q	Q	Q	Q									Q
OU-6	IR Site 26	26PZ02	Unknown	FWBZ	6/2/2014	Metals, VOCs, MNA	Active	Q								Q	Q	Q	Q	Q	Q	Q	Q	Q									Q
OU-6	IR Site 26	26PZ03	Unknown	FWBZ	6/2/2014	Metals, VOCs, MNA	Active	Q								Q	Q	Q	Q	Q	Q	Q	Q	Q									Q
OU-6	IR Site 26	26SW01	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 26	26SW02	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 26	26SW03	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 26	26SW04	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 26	26SW05	6.2 to 16.2	FIRST	NA	NA	Inactive																										
OU-6	IR Site 26	BSU1	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-6	IR Site 26	BSU2	Unknown	UNDEFINED	NA	NA	Inactive																										
OU-6	**IR Site 27**	**Status: Groundwater monitoring for VOCs (OPS, MNA for VOCs, nearing RGs)**																															
OU-6	IR Site 27	15MJ-MW1	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	15-MW1	Unknown	Shallow	5/30/2014	Metals TPH, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	15-MW2	Unknown	Shallow	6/3/2014	Metals TPH, VOCs, MNA	Active	S																									S
OU-6	IR Site 27	15-MW3	Unknown	Shallow	5/30/2014	Metals TPH, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-01	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-02	Unknown	Shallow	6/5/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-03	Unknown	Shallow	5/30/2014	Metals, VOCs	Active	S																									S
OU-6	IR Site 27	27EW-04	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-05	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S																									S
OU-6	IR Site 27	27EW-08	Unknown	Shallow	6/4/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-12	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-13	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-14	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-6	IR Site 27	27EW-15	Unknown	Shallow	5/30/2014	Metals, VOCs	Active	S																									S
OU-6	IR Site 27	27EW-16	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-17	Unknown	Shallow	5/30/2013	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-18	Unknown	Shallow	6/4/2014	Metals, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27EW-19	Unknown	Shallow	5/30/2014	Metals, VOCs	Active	S																									S
OU-6	IR Site 27	27EW-20	Unknown	Shallow	5/30/2014	Metals, VOCs	Active	S																									S
OU-6	IR Site 27	27MW01	Unknown	Shallow	6/3/2014	Metals, VOCs, MNA	Active	S																									S
OU-6	IR Site 27	27MW02	Unknown	Shallow	5/30/2014	Metals, VOCs, MNA	Active	S																									S
OU-6	IR Site 27	27MW03	Unknown	FIRST	7/13/2009	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 27	27MW04	Unknown	FIRST	3/6/2008	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 27	27MW05	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	27MW06	Unknown	Shallow	5/30/2014	Metals TPH, VOCs, MNA	Active	S							S	S	S	S	S		S	S	S										S
OU-6	IR Site 27	27MW07	6 to 16	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	27MW08	6 to 16	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	37-MJ-MW-10	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	37-MJ-MW11	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	37-MJ-MW12	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	37-MJ-MW8	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	37-MJ-MW-9	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	CA11-06	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	CA11-07	Unknown	FIRST	NA	NA	Inactive																										
OU-6	IR Site 27	CA11-08	Unknown	FIRST	NA	NA	Inactive																										
OU-6	**IR Site 28**	**Status: Remedial Action complete, ISCO, in-situ bioremediation, groundwater monitoring, institutional controls (OPS, LTM for copper, arsenic)**																															
OU-6	IR Site 28	28SW01	Unknown	FIRST	6/19/2009	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 28	28SW02	Unknown	FWBZ	6/3/2014	Metals, VOCs, MNA	Active					S[6]																					
OU-6	IR Site 28	28SW03	Unknown	FWBZ	6/4/2014	Metals, VOCs, MNA	Active					S[6]																					

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
OU-6	IR Site 28	28SW04	Unknown	FIRST	6/19/2009	Metals, VOCs, MNA	Inactive																										
OU-6	IR Site 28	28SW05	Unknown	UNDEFINED	5/29/2012	Metals, MNA	Inactive																										
OU-6	IR Site 28	28SW06	Unknown	UNDEFINED	5/30/2012	Metals	Inactive																										
OU-6	IR Site 28	28SW07	Unknown	UNDEFINED	5/29/2012	Metals	Inactive																										
OU-6	IR Site 28	28SW08	Unknown	FWBZ	6/3/2014	Metals, VOCs	Active					S[6]																					
OU-6	IR Site 28	28SW09	Unknown	UNDEFINED	5/29/2012	Metals	Inactive																										
IR Site 32	**IR Site 32**	**Status: Remedial action in progress, ISCO and institutional controls. Anticipated completion late 2015. LTM in BGMP thereafter (ROD in place)**																															
IR Site 32	IR Site 32	IR32-MW-06	Unknown	FWBZ	6/2/2014	VOCs, MNA, RAD	Active																				S						S
IR Site 32	IR Site 32	IR32-MW-07	Unknown	FWBZ	6/2/2014	VOCs, MNA, RAD	Active																				S						S
IR Site 32	IR Site 32	M005-A	2.9 to 12.9	FWBZ	6/2/2014	VOCs, RAD	Active																			S	S	S	S	S	S	S	S
IR Site 32	IR Site 32	M006-A	4 to 14	FWBZ	6/3/2014	Cyanide, Metals, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
IR Site 32	IR Site 32	M007-A	3.5 to 13.5	FWBZ	6/3/2014	Cyanide, Metals, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
IR Site 32	IR Site 32	M025-A	4 to 14	FWBZ	6/3/2014	Cyanide, Metals, PCBs, Pesticides, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
IR Site 32	IR Site 32	M031-A	2.5 to 12.5	FWBZ	6/2/2014	Cyanide, Metals, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
IR Site 32	IR Site 32	M032-A	2.5 to 12.5	FWBZ	6/3/2014	Cyanide, Metals, SVOCs, VOCs, RAD	Active																			S	S	S	S	S	S	S	S
Miscellaneous Wells and IR Sites																																	
NA	CAA-02	FS1MJ-MW-3	Unknown	FIRST	NA	NA	Inactive																										
NA	CAA-10	491MJ-MW1	Unknown	FIRST	NA	NA	Inactive																										
NA	CAA-10	491-MW1	Unknown	FIRST	NA	NA	Inactive																										
NA	CAA-10	491-MW2	Unknown	FIRST	NA	NA	Inactive																										
NA	CAA-10	491-MW3	Unknown	FIRST	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
NA	CAA-11	37-MJ-MW2	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	CAA-11	37-MJ-MW6	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	CAA-9A	584MW-1	Unknown	FIRST	NA	NA	Inactive																										
NA	CAA-9A	584MW-2	Unknown	FIRST	NA	NA	Inactive																										
NA	CAA-9A	584MW-3	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 25	M25-05	Unknown	FIRST	8/16/2011	Metals, PAHs, VOCs, MNA	Inactive																										
OU-5	IR Site 25	S-02	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 25	S-12	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 30	M25-03	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 30	S-13	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 30	S-16	Unknown	FIRST	NA	NA	Inactive																										
OU-5	IR Site 30	S-16-2	Unknown	FIRST	NA	NA	Inactive																										
IR Site 32	IR Site 32	IR32-MW-01	Unknown	UNDEFINED	12/20/2007	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
IR Site 32	IR Site 32	IR32-MW-02	Unknown	UNDEFINED	12/18/2007	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
IR Site 32	IR Site 32	IR32-MW-03	Unknown	UNDEFINED	12/18/2007	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
IR Site 32	IR Site 32	IR32-MW-04	Unknown	UNDEFINED	12/20/2007	Metals, PCBs, Pesticides, SVOCs	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						TPH, VOCs, MNA, RAD																											
IR Site 32	IR Site 32	IR32-MW-05	Unknown	UNDEFINED	12/18/2007	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA, RAD	Inactive																										
IR Site 32	IR Site 32	IR32-MW-08	Unknown	UNDEFINED	8/28/2012	VOCs, MNA, RAD	Inactive																										
IR Site 32	IR Site 32	M025-C	70 to 79.5	SECOND	5/30/2012	Cyanide, Metals, SVOCs, VOCs, RAD	Inactive																										
IR Site 32	IR Site 32	M025-E	17 to 27	FIRST	NA	NA	Inactive																										
NA	IR Site 34	MW-20	Unknown	UNDEFINED	3/1/2007	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA	Inactive																										
NA	IR Site 34	MW-23	Unknown	UNDEFINED	3/1/2007	Metals, PCBs, Pesticides, SVOCs TPH, VOCs, MNA	Inactive																										
NA	IR Site 35	130AOC23-MW-01	4 to 14	FIRST	6/4/2012	VOCs, MNA	Inactive																										
NA	IR Site 35	130AOC23-MW-02	3 to 13	FIRST	6/4/2012	VOCs, MNA	Inactive																										
NA	IR Site 35	130AOC23-MW-03	4 to 14	FIRST	6/4/2012	VOCs, MNA	Inactive																										
NA	IR Site 35	130AOC23-MW-04	4 to 14	FIRST	6/4/2012	VOCs, MNA	Inactive																										
NA	IR Site 35	13-MW-01	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	IR Site 35	13-MW-02	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	IR Site 35	13-MW-03	Unknown	FIRST	6/19/2009	SVOCs, VOCs, MNA	Inactive																										
NA	IR Site 35	23G-MW01	Unknown	UNDEFINED	4/25/2012	Metals, SVOCs TPH, VOCs	Inactive																										
NA	IR Site 35	23G-MW02	Unknown	UNDEFINED	2/24/2009	Metals TPH, VOCs	Inactive																										
NA	IR Site 35	23G-MW03	Unknown	UNDEFINED	4/25/2012	Metals, SVOCs	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
						TPH, VOCs																											
NA	IR Site 35	EBS98-DGS-PZ01	Unknown	FIRST	NA	NA	Inactive																										
NA	IR Site 35	EBS98-DGS-PZ02	Unknown	FIRST	NA	NA	Inactive																										
NA	IR Site 35	EBS98-DGS-PZ03	Unknown	FIRST	NA	NA	Inactive																										
NA	IR Site 35	MBG-1	Unknown	FIRST	NA	NA	Inactive																										
NA	IR Site 35	MBG-2	Unknown	FIRST	NA	NA	Inactive																										
OU-1	IR Site 7	M07A-08	Unknown	FIRST	9/12/2005	Metals, PAHs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 7	M07A-09	Unknown	FIRST	9/12/2005	Metals, PAHs TPH, VOCs, MNA	Inactive																										
OU-1	IR Site 7	M07A-11	Unknown	FIRST	6/17/2008	PAHs, VOCs	Inactive																										
OU-2A	IR Site 9	PEZ-S09-10	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	ANNEX-EW-2	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	CA06-01	Unknown	FIRST	3/19/2014	PAHs, SVOCs TPH, VOCs	Inactive																										
NA	N/A	CA06-02	Unknown	FIRST	3/19/2014	PAHs, SVOCs TPH, VOCs	Inactive																										
NA	N/A	CA09-01	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	CA09-02	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	CA10-01	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	CA10-02	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	CA11-03	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	CA11-04	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	CA11-05	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	M007-C	Unknown	SECOND	NA	NA	Inactive																										
NA	N/A	M008-A	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	M010-B-02	Unknown	UNDEFINED	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

								ORGANIC COMPOUNDS							NATURAL ATTENUATION PARAMETERS											RADIONUCLIDES							
OPERABLE UNIT	IR SITE	SAMPLING LOCATION	SCREEN INTERVAL (FT BGS)	WATER-BEARING ZONE	LAST YEAR SAMPLED	LAST ANALYTICAL GROUPS	WELL STATUS IN BGMP	VOCs (EPA 8260B)	*1,4-Dioxane (EPA 8270 SIM)*	SVOCs (EPA 8270C)	Pesticides (EPA 8081)	Dissolved Metals (EPA 6010B/6020A/7470A)	Hexavalent Chromium (EPA 7196A)	Cyanide (EPA 335.2)	Dissolved Gases - methane, ethane, ethene (RSK 175)	Anions - Nitrate, Nitrite, Chloride, Sulfate (EPA 300.0)	Alkalinity (EPA 310.2)	Sulfide (EPA 376.2)	Ferrous Iron by HACH	TDS (EPA 160.1)	BOD (EPA 405.1)	COD (EPA 410.4)	TOC (EPA 415.1)	DOC (ASTM 5310C)	Census DNA (qPCR)	Gross Alpha, Gross Beta (EPA 9310/900.0)	Radium-226 (EPA 9315/903.0)	Tritium (EPA 906.0)	Total Radium (EPA 904.0)	Strontium-90 (EPA 905.0)	Gamma Emitting Radionuclides; Cs-137 and Co-60 (EPA 901.1)	Uranium Isotopes (EPA 908 Modified)	WQ Parameters
NA	N/A	M011-02	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	M02-P	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	M102-A	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	M104-C	Unknown	AQUITARD	NA	NA	Inactive																										
NA	N/A	M16-05	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	MBG-4	Unknown	FIRST	NA	NA	Inactive																										
NA	N/A	MG1-05-D	Unknown	VADOSE	NA	NA	Inactive																										
NA	N/A	OU5-SG-20D	Unknown	VADOSE	NA	NA	Inactive																										
NA	N/A	OU5-SG-20S	Unknown	VADOSE	3/15/2005	NA	Inactive																										
NA	N/A	P01-01	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P01-02	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P01-03	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P02-01	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P02-02	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P02-03	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P05-01	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P05-02	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	P05-03	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	PEZ-17-01	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	PEZ-17-02	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	PEZ-17-03	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	PEZ-17-04	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	PEZ-S10B-05	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	PEZ-S10B-06	Unknown	UNDEFINED	NA	NA	Inactive																										
NA	N/A	WA-2	Unknown	UNDEFINED	NA	NA	Inactive																										

SAP WORKSHEET #18 CONTINUED

Notes:

1 Benzene and ethylbenzene only
2 Priority Pollutant metals only
3 Arsenic, cadmium, chromium, cobalt, iron, lead, manganese, nickel, thallium
4 Arsenic and iron only
5 Arsenic, copper, and nickel only
6 Arsenic and copper only
7 One sampling event will be conducted in 2014.

Acronyms and Abbreviations:

A = annually; ASTM = American Society for Testing and Materials; BGMP = Basewide Groundwater Monitoring Program; bgs = below ground surface; DNA = Deoxyribonucleic Acid; EPA = United States Environmental Protection Agency; ESD = Explanation of Significant Differences; FT = feet; FWBZ = first water bearing zone; GW = groundwater; IC = Institutional Control; IR = Installation Restoration; ISCO = in situ chemical oxidation; LTM = Long Term Monitoring; MNA = Monitored Natural Attenuation; NA = not applicable; NFA = No Further Action; OPS = Operating Properly and Successfully; OU = Operable Unit; PAH = Polycyclic Aromatic Hydrocarbon; PCB = polychlorinated biphenyl; Q = quarterly; qPCR = Quantitative Real-Time Polymerase Chain Reaction; RA = Remedial Action; RAD = radiological; RD = Remedial Design; RG = Remedial Goal; ROD = Record of Decision; SA = semiannually; SIM = Selective Ion Monitoring; SVOC = semi-volatile organic compound; SWBZ = second water bearing zone; TPH = total petroleum hydrocarbon; VA = Veterans Affairs; VOC = volatile organic compound; WQ = water quality

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SAP WORKSHEET #19 ANALYTICAL SOP REQUIREMENTS TABLE

Table 19-1. Analytical SOP Requirements

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers	Minimum Sample Volume	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
Soil Gas	Fixed Gases (CO, O_2, N_2, and CO_2)	U.S. EPA TO-03	1-liter Summa Canister[1]	Up to 1 liter	None	30 days from sampling
Soil Gas	VOCs including: Methane, Ethane, Ethene	Method TO-15 WS-MSA-0015	6-liter Summa™ Canister	Up to 6 liters	None	30 days from sampling
Groundwater	VOCs	U.S. EPA 5030B/8260B WS-MS-0007	3 x 40 mL glass vials with PTFE lined septum and an open screw cap	40 ml	No headspace; pH<2; Cool ≤6°C; protect from light	Analysis within 14 days from sampling
Groundwater	1,4-Dioxane	U.S. EPA 8270C-SIM	1-liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C	Extraction within 7 days from sampling/40 days from extraction
Groundwater	SVOCs	U.S. EPA 3510C/8270D WS-OP-0001, WS-MS-0005	1-liter amber glass with PTFE lined screw cap	1 liter	Cool to 4 ± 2 C	Extraction within 7 days from sampling/ Analysis 40 days from extraction
Groundwater	TPH-E	U.S. EPA 8015B	1-liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C	Extraction within 7 days from sampling/ analysis 40 days from extraction
Groundwater	TPH-P	U.S. EPA 8015B	40 ml glass vials with PTFE lined septum and an open screw cap	40 mL	Cool ≤6°C	Analysis within 14 days from sampling
Groundwater	Alkalinity	U.S. EPA 310.1	250 mL polypropylene container	250 mL	Cool ≤6°C	Analysis within 14 days from sampling
Groundwater	Sulfide	U.S. EPA 376.2	250 mL polypropylene container	250 mL	NaOH/ZnAc; Cool ≤6°C	Analysis within 7 days from sampling

SAP WORKSHEET #19 CONTINUED

Table 19-1. Analytical SOP Requirements (continued)

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers	Minimum Sample Volume	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
Groundwater	COD	U.S. EPA 410.4/ WS-WC-0040	1 x 500-mL HDPE	100 mL	Cool to 4 ± 2 C; H_2SO_4 to pH < 2	28 days
Groundwater	Methane, Ethane, Ethene	RSK 175	40-mL glass vials with PTFE lined septum and an open screw cap	40 mL	Acidified to a pH <2 with HCl; Cool ≤6°C	Analysis within 14 days from sampling
Groundwater	Pesticides	U.S. EPA 3510C/8081A WS-OP-0002, WS-GC-0001	1-liter amber glass with PTFE lined screw cap	1 liter	Cool to 4 ± 2 C	Extraction within 7 days from sampling/ analysis 40 days from extraction
Groundwater	PCBs	U.S. EPA 3550, U.S. EPA 8082	1-liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C, protect from light	Extraction within 7 days from sampling/ analysis 40 days from extraction
Groundwater	PAHs	U.S. EPA 8310	1-liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C	Extraction within 7 days from sampling/analysis 40 days from extraction
Groundwater	TDS	U.S. EPA150.1	1000-mL polypropylene container	125 mL	Cool ≤6°C	Analysis within 7 days from sampling
Groundwater	Cyanide	U.S. EPA 9012A/335.2	500-mL polypropylene container	250 mL	NaOH	Analysis within 14 days from sampling
Groundwater	Dissolved Metals	U.S. EPA 3005A or 3010A/6020 WS-IP-0001, WS-MT-0001	1 x 100-mL HDPE	100 mL	HNO_3 to pH <2, Cool to 4 ± 2 C	Analysis within 180 days from sampling
Groundwater	Mercury	U.S. EPA 7470A WS-MT-0005	1 x 100-mL HDPE	100 mL	HNO_3 to pH <2, Cool to 4 ± 2 C	Analysis within 28 days from sampling
Groundwater	Anions (Chloride, Nitrate, Nitrite, and Sulfate)	U.S. EPA 300 / WS-LC-0009	1 x 500-mL HDPE	100 mL	Cool to 4 ± 2 C	48 hours (nitrate/nitrite/phosphate/28 days (chloride, sulfate, fluoride, bromide)

SAP WORKSHEET #19 CONTINUED

Table 19-1. Analytical SOP Requirements (continued)

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers	Minimum Sample Volume	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
Groundwater	Hexavalent Chromium (Cr+6)	U.S. EPA 7196A / WS-WC-0020	1 x 500-mL HDPE or Glass	250 mL	Cool to 4 ± 2 C	Analysis within 24 hours from sampling
Groundwater	VOCs	EPA 8260C	3 x 40 ml glass vials with PTFE lined septum and an open screw cap	120 ml	No headspace; HCl pH<2; Cool ≤6°C; protect from light	Analysis within 14 days from sampling
Groundwater	1,4-Dioxane	EPA 8260C-SIM	1 Liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C	Extraction within 7 days from sampling/40 days from extraction
Groundwater	SVOCs	EPA 8270D	1 Liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C	Extraction within 7 days from sampling/ Analysis 40 days from extraction
Groundwater	TPH-E	EPA 8015B	1 Liter amber glass with PTEE lined screw cap	1 liter	HCl; Cool ≤6°C	Extraction within 7 days from sampling/ Analysis 40 days from extraction
Groundwater	TPH-P	EPA 8015B	40 ml glass vials with PTFE lined septum and an open screw cap	40 mL	HCl, Cool ≤6°C	Analysis within 14 days from sampling
Groundwater	Alkalinity	EPA 310.1	250 mL polypropylene container	250 mL	Cool ≤6°C	Analysis within 14 days from sampling
Groundwater	Sulfide	EPA 376.1	250 mL polypropylene container	250 mL	NaOH/ZnAc; Cool ≤6°C	Analysis within 7 days from sampling
Groundwater	Methane, Ethane, Ethene	RSK 175	40 ml glass vials with PTFE lined septum and an open screw cap	40 mL	Acidified to a pH <2 with HCl; Cool ≤6°C	Analysis within 14 days from sampling

SAP WORKSHEET #19 CONTINUED

Table 19-1. Analytical SOP Requirements (continued)

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers	Minimum Sample Volume	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
Groundwater	Pesticides	EPA 8081B	1 Liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C	Extraction within 7 days from sampling/ Analysis 40 days from extraction
Groundwater	PCBs	EPA 8082A	1 Liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C, protect from light	Extraction within 7 days from sampling/ Analysis 40 days from extraction
Groundwater	PAHs	EPA 8270D SIM	1 Liter amber glass with PTFE lined screw cap	1 liter	Cool ≤6°C	Extraction within 7 days from sampling/ Analysis 40 days from extraction
Groundwater	TDS	EPA160.1	125 mL polypropylene container	125 mL	Cool ≤6°C	Analysis within 7 days from sampling
Groundwater	Cyanide	EPA 9012B	250 mL polypropylene container	250 mL	NaOH	Analysis within 14 days from sampling
Groundwater	Dissolved Metals	EPA 6020A	polypropylene container	100 ml	Acidified to a pH <2 with HNO3	Analysis within 180 days from sampling
Groundwater	Mercury	EPA 7470A	polypropylene container	100 ml	Acidified to a pH <2 with HNO3	Analysis within 28 days from sampling
Groundwater	Anions (Chloride, Nitrate, Nitrite, and Sulfate)	EPA 300.0	Glass or polypropylene container	100 ml	Cool ≤6°C	Analysis within 28 days (48 hours for nitrate) from sampling
Groundwater	Hexavalent Chromium (Cr+6)	EPA 7196A	125 ml polypropylene container	10 ml	Adjust the pH to 9-9.5 by ammonium sulfate buffer solution; Cool ≤6°C	Analysis within 24 hours from sampling
Groundwater	Gross Alpha	EPA 9310/900.0	polypropylene container	1 liter of water	Acidified to a pH <2 with HNO3	Analysis within 48 days from sampling
Groundwater	Gross Beta	EPA 9310/900.0	polypropylene container	1 liter of water	Acidified to a pH <2 with HNO3	Analysis within 48 days from sampling

SAP WORKSHEET #19 CONTINUED

Table 19-1. Analytical SOP Requirements (continued)

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers	Minimum Sample Volume	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
Groundwater	Tritium	EPA 906.0	250 mL glass amber bottle	500 mL	none	Analysis within 48 days from sampling
Groundwater	Gamma Emitting Radionuclides (Cs-137 and Co-60)	EPA 901.1	polypropylene container	1 liter of water	Acidified to a pH <2 with HNO3	Analysis within 48 days from sampling
Groundwater	Radium-226	EPA 903.0	polypropylene container	1 liter of water	Acidified to a pH <2 with HNO3	Analysis within 48 days from sampling
Groundwater	Uranium Isotopes	DOE A-01-R MOD	polypropylene container	1 liter of water	Acidified to a pH <2 with HNO3	Analysis within 48 days from sampling
Groundwater	Total Radium	EPA 904.0	polypropylene container	1 liter of water	Acidified to a pH <2 with HNO3	Analysis within 48 days from sampling
Groundwater	Strontium-90	EPA 905.0	1 liter bottle	1 liter of water	Acidified to a pH <2 with HNO3	Analysis within 48 days from sampling
Groundwater/IDW	Gross Alpha/Beta	U.S. EPA 900.0 SOP ST-RD-0403	One 500-mL, poly	1 L	HNO_3 pH < 2	180 days
Groundwater/IDW	Tritium	U.S. EPA 906.0 SOP ST-RD-0302	One 500-mL,Glass	1 L	None	180 days
Groundwater/IDW	Gamma Spectroscopy	U.S. EPA 901.1 SOP ST-RD-0102	One 1-liter, poly	1 L	HNO_3 pH < 2	180 days
Groundwater/IDW	Radium-226	U.S. EPA 903.0 SOP ST-RD-0403	One 1-liter, poly	1 L	HNO_3 pH < 2	180 days
Groundwater/IDW	Isotopic Uranium	DOE A-01-R MOD SOP ST-RC-0210	One 1-liter, poly	1 L	HNO_3 pH < 2	180 days
Groundwater/IDW	Radium-228	U.S. EPA 904.0 SOP ST-RD-0403	One 1-liter, poly	1 L	HNO_3 pH < 2	180 days
Groundwater/IDW	Strontium-90	U.S. EPA 905.0 SOP ST-RD-0403	One 1-liter, poly	1 L	HNO_3 pH < 2	180 days

Notes:

1. A single summa canister can be collected at one location for TO-03 and TO-15 analysis.

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SAP WORKSHEET #20 FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates[2]	No. of MS/MSDs	No. of Field Blanks[2]	No. of Temp. Blanks	No. of Equip. Blanks[2]	No. of VOA Trip Blanks	Anticipated No. of Samples to Lab
Soil Gas	Fixed Gases; Methane, Ethane, Ethene	9	1	0	0	0	0	0	10
Groundwater	VOCs; 1,4-Dioxane; General Chemistry Suite; Sulfide; Radionuclide Suite; SVOCs; Dissolved Metals; Hexavalent Chromium; Pesticides; Cyanide; TDS	Variable [1]	10% of primary samples	5% of primary samples	once per source water	one per cooler	one per day per sampling team	one per cooler	Variable [1]

Notes:

1. Numbers of BGMP wells subject to groundwater sampling vary on a quarterly basis.
2. Field Duplicates, Field Blanks, Source Blanks, and Equipment Blanks will only be collected for analytical suite 1 as defined in Worksheet #12 - TPH, VOCs, 1,4-dioxane, SVOCs, PAHs, pesticides, PCBs, metals, cyanide, and radionuclides

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SAP WORKSHEET #21 PROJECT SAMPLING SOP REFERENCES TABLE

All project-specific tasks and procedures are detailed within SAP Worksheets #14 and #17.

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SAP WORKSHEET #22 FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

Field Equipment [1]	Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference	Comments
PID	Visual Inspection	Daily, prior to sampling activity	Within manufacturer's recommended values	Operator correction or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–
	Maintenance and Charging	Daily, prior to sampling activity. Maintenance check at end of day	Within manufacturer's recommended values	Operator correction or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–
	Calibration	Daily, prior to sampling activity and as needed	Within manufacturer's recommended values	Recalibrate until acceptable range or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–
	Testing	Daily, during sampling activity	Within manufacturer's recommended values	Operator correction or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–
Water Quality Meter	Visual Inspection	Daily, prior to sampling activity	Within manufacturer's recommended values	Operator correction or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–
	Maintenance and Charging	Daily, prior to sampling activity. Maintenance check at end of day	Within manufacturer's recommended values	Operator correction or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–
	Calibration	Daily, prior to sampling activity and as needed	Within manufacturer's recommended values	Recalibrate until acceptable range or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–
	Testing	Daily, during sampling activity	Within manufacturer's recommended values	Operator correction or return to manufacturer for repair.	Ulf Richter	Manufacturer's operating instructions	–

Notes:

1. Rental equipment and instruments are frequently used in the field by the MMEC Group. The rental firms will be responsible for the proper care, maintenance, and repair of these items, and for tracking and documenting equipment and instrument maintenance and repairs.

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SAP WORKSHEET #23 ANALYTICAL SOP REFERENCES TABLE

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-MT-0001	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry [Methods 6020 and 6020A] (Revision 3.7, Effective 8/26/2014)	Definitive	Water & Soil Metals	ICPMS	TestAmerica Sacramento	N
WP-IP-0001	Acid Digestion of Aqueous Samples by SW-846 [Methods 3005A, 3010A], (revision 5.3, Effective 03/28/2014)	Definitive	Water Metals	NA	TestAmerica Sacramento	N
WS-IP-0002	Acid Digestion of Soils, SW-846 Method 3050B (Revision 5.3, Effective 1/19/2012, Reviewed 04/14/2014)	Definitive	Soil Metals	NA	TestAmerica Sacramento	N
WS-GC-0001	Organochlorine Pesticides by Gas Chromatography Based on SW-846 Methods 8000B/8000C, 8081A/8081B, Method 608 and Compendium Methods TO-4A and TO-10A (Revision 5.1 Effective 8/22/2014)	Definitive	Water & Soil Pesticides	GC-ECD	TestAmerica Sacramento	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-OP-0002	Extraction and Cleanup of Organic Compounds from Waters and Soils, based on SW-846 3500 Series and 3600 Series Methods for Analysis by Methods 8081A and 8082 (Revision 4.1 Effective 5/2/2014)	Definitive	Water & Soil Pesticides	NA	TestAmerica Sacramento	N
WS-GC-0002	Chromatographic Analysis Based on SW-846 Methods 8000B/8000C, 8082/8082A, Method 608, and Compendium Methods TO-4A and TO-10A (Revision 5.0, Effective 04/15/2014)	Definitive	Water & Soil PCBs	GC-ECD	TestAmerica Sacramento	N
WS-OP-0002	Extraction and Cleanup of Organic Compounds from Waters and Soils, based on SW-846 3500 Series and 3600 Series Methods for Analysis by Methods 8081A and 8082 (Revision 4.1 Effective 5/2/2014)	Definitive	Water & Soil PCBs	NA	TestAmerica Sacramento	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-MS-0007	Determination of Volatile Organics and Total Purgeable Petroleum Hydrocarbons by GC/MS {Methods 8260B, 8260C, 624, Cal-LUFT and AK101), Revision 5.0, Effective 04/15/2014	Definitive	Groundwater Volatiles	GC/MS	TestAmerica Sacramento	N
WS-MS-0007	Determination of Volatile Organics and Total Purgeable Petroleum Hydrocarbons by GC/MS {Methods 8260B, 8260C, 624, Cal-LUFT and AK101), Revision 5.0, Effective 04/15/2014	Definitive	Soil Volatiles	GC/MS	TestAmerica Sacramento	N
WS-MS-0005	Semivolatile Organic Compounds (Base/Neutral and Acids) by Gas Chromatography/Mass Spectroscopy (GC/MS) [Methods 8270C, 8270D, 625 & TO-13A] Revision 5.0, Effective 4/15/2014	Definitive	Groundwater Semivolatiles	GC/MS	TestAmerica Sacramento	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-OP-0001	Extraction of Semivolatile Organic Compounds by Method 8270C, Based on SW-846 3500 Series and 3600 Series, and PAH-SIM by Internal Standard and Isotope Dilution Procedures, Revision 4.0, Effective 08/09/2013	Definitive	Groundwater Semivolatiles	Preparation	TestAmerica Sacramento	N
WS-MS-0005	Semivolatile Organic Compounds (Base/Neutral and Acids) by Gas Chromatography/Mass Spectroscopy (GC/MS) [Methods 8270C, 8270D, 625 & TO-13A] Revision 5.0, effective 4/15/2014	Definitive	Soil Semivolatiles	GC/MS	TestAmerica Sacramento	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-OP-0001	Extraction of Semivolatile Organic Compounds by Method 8270C, Based on SW-846 3500 Series and 3600 Series, and PAH-SIM by Internal Standard and Isotope Dilution Procedures, Revision 4.0, Effective 08/09/2013	Definitive	Soil Semivolatiles	Preparation	TestAmerica Sacramento	N
WS-WC-0028	Determination of Alkalinity, Conductivity, pH and Fluoride (Methods Sm 4500 H+ B, SM 2320B, EPA 9214, EPA 9040B, EPA 9040C, EPA 120.1, SM2150B); Revision 4.4, Effective 03/28/2014	Definitive	Aqueous Alkalinities	Autotitrator	TestAmerica Sacramento	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-WC-0009	Determination of Anions by Ion Chromatography (Method 300.0A & 9056); Revision 3.2, Effective 03/21/2014	Definitive	Anion Waters	Ion Chromatograph	TestAmerica Sacramento	N
WS-WC-0049	Deionized Leaching Procedure for General Chemistry Analysis, Revision 4.2, Effective 6/28/2013	Definitive	Anion Solids	NA	TestAmerica Sacramento	N
WS-WC-0020	Determination of Hexavalent Chromium by Manual Colorimetric Method (Method SW-46/ 7196A); Revision 7.5, Effective 10/21/2013	Definitive	Aqueous and Solid Chromium VI	Spectrophotometer	TestAmericaSacramento	N
WS-MT-0005	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A and MCAWW 245.1 (Revision 5.5, Effective 07/11/2014)	Definitive	Water Mercury	CVAA	TestAmerica Sacramento	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-MT-0007	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption (Revision 5.3, Effective 1/7/2014)	Definitive	Soil Mercury	CVAA	TestAmerica Sacramento	N
WS-MS-0008	Determination of Polycyclic Aromatic Hydrocarbons (PAH) by GC/MS-SIM Internal Standard Technique (Revision 2.5, Effective 6/6/2014)	Definitive	Soil and Water PAHs	GC/MS	TestAmerica Sacramento	N
WS-OP-0001	Extraction of Semivolatile Organic Compounds by Method 8270C, Based on SW-846 3500 Series and 3600 Series, and PAH-SIM by Internal Standard and Isotope Dilution Procedures, Revision 4.1, Effective 08/22/2014	Definitive	Soil and Water PAHs	Preparation	TestAmerica Sacramento	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WS-WC-0040	Determination of Chemical Oxygen Demand (Method EPA 410.4); Revision 3, Effective 1/30/2009	Definitive	Aqueous COD	Spectrophotometer	TestAmerica Sacramento	N
WS-MSA-0015	Determination of Low-Level Volatile Organics in Ambient/Indoor Whole Air Samples using GC/MS Scan Mod [Methods EPA TO-14A and EPA TO-15], (Revision 1.1, Effective 4/22/2013)	Definitive	Air Volatiles	GC/MS	TestAmerica Sacramento	N
ST-RD-0102	GammaVision Analysis Rev. 12, 04/16/14	Definitive	Water Gamma Spectroscopy	Gamma Spectrometer	TestAmerica –St. Louis	N
ST-RC-0210	Alpha Spectroscopy Analysis Rev. 11, 08/11/13	Definitive	Water Isotopic Uranium	Alpha Spectrometer	TestAmerica –St. Louis	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
ST-RD-0403	Low Background Gas Flow Proportional Counting System Analysis Rev. 14, 09/12/13	Definitive	Water Alpha/Beta, Ra-226, Ra-228, Sr-90	Gas Proportional Counter	TestAmerica –St. Louis	N
ST-RD-0302	Liquid Scintillation Counter Analysis Rev. 14, 05/08/13	Definitive	Water Tritium	Liquid Scintillation	TestAmerica –St. Louis	N
ST-MS-0002	Determination of Volatile Organics by GC/MS Rev. 23, 12/01/14	Definitive	Water/ Volatiles	GC/MS	TestAmerica –St. Louis	N
ST-MS-0002	Determination of Volatile Organics by GC/MS Rev. 23, 12/01/14	Definitive	Water/ 1,4-Dioxane	GC/MS	TestAmerica –St. Louis	N
ST-MS-0001	GC/MS Semi-Volatile Analysis Rev.19, 12/01/14	Definitive	Water/ Semi-Volatiles	GC/MS	TestAmerica –St. Louis	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
ST-MS-0001	GC/MS Semi-Volatile Analysis Rev.19, 12/01/14	Definitive	Water/ PAHs	GC/MS	TestAmerica –St. Louis	N
ST-GC-0016	Pesticide Gas Chromatographic Analysis Rev. 18, 12/01/14	Definitive	Water/ Pesticides	GC-ECD	TestAmerica –St. Louis	N
ST-GC-0015	PCB GC Analysis Rev. 15, 12/01/14	Definitive	Water/ PCBs	GC-ECD	TestAmerica –St. Louis	N
ST-GC-0005	Extractable Total Petroleum Hydrocarbons by GC-FID Rev. 19, 06/02/14	Definitive	Water/ TPH-DRO/MRO	GC-FID	TestAmerica –St. Louis	N
ST-GC-0014	Aromatic Volatiles and Volatile Petroleum Hydrocarbons by GC PID/FID Rev. 12, 06/02/14	Definitive	Water/ TPH-GRO	GC-PID/FID	TestAmerica –St. Louis	N
ST-GC-0019	Analysis of Dissolved Gases in Groundwater Rev. 2, 06/02/14	Definitive	Water/ RSK-175	GC-FID	TestAmerica –St. Louis	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
ST-MT-0001	Analysis of Metals by Inductively Coupled Plasma Mass Spectroscopy Rev. 22, 08/27/13	Definitive	Water/ Metals	ICP-MS	TestAmerica –St. Louis	N
ST-MT-0005	Preparation and Analysis of Mercury in Aqueous Samples by CVAA, Rev. 15, 08/25/14	Definitive	Water/ Mercury	Cold Vapor AA	TestAmerica –St. Louis	N
ST-WT-0002	Cyanide Analysis by the Technicon TRAACS 800 Autoanalyzer, Rev. 17, 01/26/2015	Definitive	Water/ Cyanide	TRAACS 800	TestAmerica –St. Louis	N
ST-WT-0012	Analysis of Sulfide, Rev. 13, 10/31/2014	Definitive	Water/ Sulfide	Titration	TestAmerica –St. Louis	N
ST-WT-0019	Alkalinity in Water and Soil, Rev. 15, 07/26/2013	Definitive	Water/ Alkalinity	Titration	TestAmerica –St. Louis	N
ST-WT-0028	Analysis of Anions by Ion Chromatography, Rev. 21, 06/02/2014	Definitive	Water/ Anions	Ion Chromatography	TestAmerica –St. Louis	N
ST-WT-0033	Hexavalent Chromium (Colorimetric), Rev. 18, 07/30/2013	Definitive	Water/ Hexavalent Chromium	Spectrophotometer	TestAmerica –St. Louis	N

SAP WORKSHEET #23 CONTINUED

Lab SOP Number	Title, Revision Date, and/or Number [1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
ST-WT-0036	Determination of Solids in Water and Wastes, Rev. 11, 08/08/2013	Definitive	Water/ TDS	Balance	TestAmerica –St. Louis	N
ST-RD-0102	GammaVision Analysis, Rev. 12, 04/16/2014	Definitive	Water/ Gamma Spec	Gamma Spec	TestAmerica –St. Louis	N
ST-RD-0210	Alpha Spectroscopy Analysis, Rev. 11, 08/21/02013	Definitive	Water/ Isotopic Uranium	Alpha Spec	TestAmerica –St. Louis	N
ST-RD-0302	Liquid Scintillation Counter Analysis, Rev. 14, 05/08/2013	Definitive	Water/ Tritium	Liquid Scintillation	TestAmerica –St. Louis	N
ST-RD-0403	Low Background Gas Flow Proportional Counting (GFPC) System Analysis, Rev. 15, 01/19/2015	Definitive	Water/ Gross Alpha/Beta; Ra-226/228, Sr-90	Gas Flow Proportional Counter	TestAmerica –St. Louis	N

Notes:

1. The laboratory SOP is included in Attachment 3.

SAP WORKSHEET #24 ANALYTICAL INSTRUMENT CALIBRATION TABLE

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Gas Flow Proportional Counter	Plateau generation and/or verification	Annual	Plot efficiencies vs masses	Recalibrate	TestAmerica – St. Louis Group Leader	ST-RD-0403
	Discriminator setting		Calculate equation of curve – degree ≤3	Instrument maintenance		
	Initial long background count		Remove outliers >15% deviation from theoretical values but not more than 20% of total points	Consult with Technical Director		
	Mass attenuated efficiency calibration		Calculate coefficient of determination (R^2). R^2 must be ≥0.9			
	Eight source dual/single calibration curves		Verify calibration with second source standard count – must be within 30% of true value and mean across all detectors <10%			

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Gamma Spectrometer	Energy calibration	Annual	For Energy and FWHM calibration	Recalibrate	TestAmerica – St. Louis Group Leader	ST-RD-0102
	FWHM calibration		Within 0.5% or 0.1 KeV for all calibration points	Instrument maintenance		
Gamma Spectrometer	Background	Monthly	Within 8% for all calibration points. Verify with second source that always contains at least Am-241, Co-60, and Cs-137. Must be ± 10%D for each nuclide. For Background, acceptance criterion is 12 hours.	Consult with Technical Director		

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Alpha Spectrometer	Energy calibration	Monthly	Three isotopes in 3–6 MeV range all within ± 40 KeV of expected value	Recalibrate	TestAmerica – St. Louis Group Leader	ST-RC-0210
	Efficiency calibration and background check		>20%	Instrument maintenance		
	Subtraction spectrum		Ultra Low Level: < 2 CPM	Consult with Technical Director		
	Pulser check and background check	Daily	Low Level: < 2-4 CPM. Routine Level: < 4-10 CPM. High Level: < 10-20 CPM. Pulser energy, peak centroid, peak resolution, peak area, calibration and background must pass statistical "boundary" out-of-range test	If background check is > 20 CPM, then detector requires maintenance		

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Liquid Scintillation Counter	Statistical baseline for C-14 and H-3 backgrounds and efficiencies	Startup or long-term use	Developed with statistical limits at time of startup	Recalibrate	TestAmerica – St. Louis Group Leader	ST-RD-0302
	Check of C-14 and H-3 backgrounds and efficiencies	Daily	Within 3 sigma of baseline established at startup or when re-established	Instrument maintenance		
	Quench curve for specific nuclide	Annual	Second source verification ± 10% of true value	Consult with Technical Director		
GC-ECD	Breakdown Check (Pesticides only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% each for DDT and Endrin	Evaluate standard, chromatography, and detector response. If problem (e.g., active sites on column, dirty inlet) indicated, correct as appropriate, then repeat breakdown check.	Lab Manager/ Analyst	WS-GC-0001 WS-GC-0002 ST-GC-0015 ST-GC-0016

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GC-ECD	Minimum five-point initial calibration for single eluting target analytes. Single -level initial calibration for multi-component analytes (i.e., toxaphene, technical chlordane). If identified as present, reanalysis and quantitation against a multi-level calibration is performed.	Initial calibration after instrument set-up and after initial calibration verification (ICV)/ continuing calibration verification (CCV) failure, prior to sample analysis	One of the options below: (1) RSD for each analyte ≤ 20%; (2) Linear least squares regression: $r^2 \geq 0.99$; (3) non-linear regression: COD (r^2) ≥ 0.99, minimum of 6 points for second order	Evaluate standards, chromatography, and detector response. If problem found with above, correct as appropriate, then repeat initial calibration.	Lab Manager/ Analyst	WS-GC-0001 WS-GC-0002 ST-GC-0015 ST-GC-0016
GC-ECD	Retention Time Window Position Establishment	Once per initial calibration (ICAL), and at the beginning of the analytical sequence, for each analyte and surrogate	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial continuing calibration verification (CCV).	NA	Analyst	WS-GC-0001 WS-GC-0002 ST-GC-0015 ST-GC-0016

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GC-ECD	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within established RT windows, and within ± 20% of the expected value from the ICAL	Evaluate data. If problem (e.g., concentrated standard, plugged injector needle) found, correct, then repeat second source verification. If still fails, repeat initial calibration.	Lab Manager/ Analyst	WS-GC-0001 WS-GC-0002 ST-GC-0015 ST-GC-0016

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GC-ECD	Daily calibration verification	Prior to sample analysis, after every 10 field samples, and at the end of the sequence. CCVs for multi-eluting pesticides (i.e., Toxaphene, Chlordane) are required only before sample analysis, unless the analyte is detected in the associated samples.	All reported analytes and surrogates within established RT windows, and within ± 20% of the expected value from the ICAL	Evaluate standard - if response is elevated and associated samples are non-detect for that analyte, narrate. If ending CCV, and second analysis of samples due to non-compliant ending CCV, narrate. Otherwise, recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Corrective actions include: evaluate standard, chromatography, and detector response. Correct as appropriate (instrument maintenance, remake standards), then repeat initial calibration.	Lab Manager/ Analyst	WS-GC-0001 WS-GC-0002 ST-GC-0015 ST-GC-0016

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Colorimetric Analyzer 9012A or 9012B	Initial calibration (minimum five point calibration)	Initial daily calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$	Correct problem then repeat initial calibration	Lab Manager/ Analyst	DV-WC-0083 ST-WT-0002
Colorimetric Analyzer			($r \geq 0.995$ for linear regression)			
Colorimetric Analyzer	Distillation Verification	Prepared per prep batch (cyanide only)	±10% of undistilled standard value for high distilled standard	Re-distill and re-analyze all associated samples. Correct problem. Rerun ICV. If that fails, repeat ICAL.	Lab Manager/ Analyst	DV-WC-0083 ST-WT-0002
Colorimetric Analyzer			±15% of undistilled standard value for low distilled standard			
Colorimetric Analyzer	Second-source calibration verification (ICV)	Immediately following initial daily calibration	±10% of true value	Correct problem then repeat initial calibration	Lab Manager/ Analyst	DV-WC-0083 ST-WT-0002

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Colorimetric Analyzer	CCV	After every 10 field samples and at the end of the analysis sequence	±10% of true value. Within + 10% of non-distilled standard value	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment with written approval from the client. Corrective Action: Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/ Analyst	DV-WC-0083 ST-WT-0002

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Autotitrator	Minimum three point calibration with buffers of known pH	Initial calibration prior to sample analysis	r ≥ 0.995	Evaluate buffers and instrument response. If problem found with above, correct as appropriate, then repeat initial calibration.	Lab Manager/ Analyst	WS-WC-0028 ST-WT-0012 ST-WT-0019
Autotitrator	Initial calibration verification (ICV, Second Source)	Immediately following ICAL	All results within ± 10% of the expected value	Evaluate data. If problem (e.g., concentrated standard, plugged lines) found, correct, then repeat second source verification. If still fails, repeat initial calibration.	Lab Manager/ Analyst	WS-WC-0028 ST-WT-0012 ST-WT-0019
Autotitrator	Calibration Blank (ICB/CCB)	Immediately following ICV (ICB) and immediately following CCV (CCB)	Result within ± RL from zero	Evaluate data. If problem found (e.g. contaminated lines or solution), correct, then repeat. If still fails, investigate further and repeat initial calibration. Repeat all samples since last successful calibration blank.	Lab Manager/ Analyst	WS-WC-0028 ST-WT-0012 ST-WT-0019
Autotitrator	Daily calibration verification	Prior to sample analysis, after every 10 field samples, and at the end of the sequence	All results within ± 10% of the expected value	Evaluate standard and response. If problem found with above, correct as appropriate, then repeat CCV. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration verification.	Lab Manager/ Analyst	WS-WC-0028 ST-WT-0012 ST-WT-0019

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Ion Chromatograph	Retention Time Window Width Establishment	After method set-up and following major maintenance (e.g., column change)	Width is ± 3 times standard deviation for each analyte based on analyses over a 24-hour period	NA	Lab Manager/ Analyst	WS-WC-0009 ST-WT-0028
Ion Chromatograph	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit (ICAL)	Initial calibration prior to sample analysis	COD (r^2) ≥ 0.99, minimum of 4 calibration levels and a reagent blank	Evaluate standards, chromatography, and detector response. If problem found with above, correct as appropriate, then repeat initial calibration.	Lab Manager/ Analyst	WS-WC-0009 ST-WT-0028
Ion Chromatograph	ICV	Immediately following ICAL.	All project analytes within ± 10% of the expected value from the ICAL and within established RT windows	Evaluate data. If problem (e.g., concentrated standard, plugged injector needle) found, correct, then repeat second source verification. If still fails, repeat initial calibration.	Lab Manager/ Analyst	WS-WC-0009 ST-WT-0028

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Ion Chromatograph	Retention Time Window Position Establishment	Daily, prior to use	On days when ICAL is performed, set using the mid-point standard of the ICAL. If ICAL not performed, use initial CCV.	NA	Lab Manager/ Analyst	WS-WC-0009 ST-WT-0028
Ion Chromatograph	Daily calibration verification	Prior to sample analysis, after every 10 field samples, and at the end of the sequence	All project analytes within ± 10% of the expected value from the ICAL and within established RT windows	Evaluate standard - if response is elevated and associated samples are non-detect for that analyte, narrate. Otherwise, recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/ Analyst	WS-WC-0009 ST-WT-0028

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Spectrophotometer	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit	Daily calibration prior to sample analysis	Linear least squares regression: $r^2 \geq 0.99$	Evaluate standards, and spectrophotometer response. If problem found with above, correct as appropriate, then repeat initial calibration.	Lab Manager/ Analyst	WS-WC-0020 ST-WT-0033
Spectrophotometer	Initial calibration verification (ICV, Second Source)	Immediately following ICAL	Calculated concentration within ± 10% of the expected value from the ICAL	Evaluate data. If problem (e.g., concentrated standard, incorrectly prepared standard) found, correct, then repeat second source verification. If still fails, repeat initial calibration.	Lab Manager/ Analyst	WS-WC-0020 ST-WT-0033
Spectrophotometer	Calibration Blank (ICB/CCB)	Immediately following ICV (ICB) and immediately following CCV (CCB)	Result within ± RL from zero.	Evaluate data. If problem found (e.g. contaminated cuvet or solution), correct, then repeat. If still fails, investigate further and repeat initial calibration. Repeat all samples since last successful calibration blank.	Lab Manager/ Analyst	WS-WC-0020 ST-WC-0033

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Spectrophotometer	Daily calibration verification	After every 15 field samples, and at the end of the sequence	Calculated concentration within ± 10% of the expected value from the ICAL	Evaluate standard - if response is elevated and associated samples are non-detect for that analyte, narrate. If ending CCV, and second analysis of samples due to non-compliant ending CCV, narrate. Otherwise, recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Corrective actions include: evaluate standard, chromatography, and detector response. Correct as appropriate (instrument maintenance, remake standards), then repeat initial calibration.	Lab Manager/ Analyst	WS-WC-0020 ST-WC-0033

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
CVAA	Initial calibration (ICAL) per manufacturer's instructions, with a minimum of five standards and a calibration blank	Initial calibration prior to sample analysis	Correlation coefficient >0.995; accepted if the ICV passes	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat initial calibration.	Lab Manager/ Analyst	WS-MT-0005 WS-MT-0007 ST-MT-0005
CVAA	Second-source ICV, prepared at the calibration midpoint	Once per initial calibration	Less than 10% difference from IC for all target analytes	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration.	Lab Manager/ Analyst	WS-MT-0005 WS-MT-0007 ST-MT-0005

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
CVAA	CCV, same source as IC	Following IC, after every 10 samples and the end of the sequence	Less than 10% difference from IC for all target analytes	Evaluate standard - if response is elevated and associated samples are non-detect for that analyte, narrate. If ending CCV, and second analysis of samples due to non-compliant ending CCV, narrate. Otherwise, recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Corrective actions include: evaluate standard, chromatography, and detector response. Correct as appropriate (instrument maintenance, remake standards), then repeat initial calibration.	Lab Manager/ Analyst	WS-MT-0005 WS-MT-0007 ST-MT-0005
GC/MS	Check of mass tuning	Prior to ICAL and at the beginning of each 12-hour period.	Method or instrument-specific tuning criteria	Retune instrument and verify.	Lab Manager/ Analyst	WS-MS-0005 WS-MS-0007 WS-MS-0008 ST-MS-0001 ST-MS-0002

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GC/MS	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)	As needed	One of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Verify standard solutions still valid, perform instrument maintenance as needed, then repeat the ICAL.	Lab Manager/ Analyst	WS-MS-0005 WS-MS-0007 WS-MS-0008 ST-MS-0001 ST-MS-0002
GC/MS	Second-source calibration verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within ± 20% of true value..	Correct problem, and verify second source standard. Rerun verification. If still fails, repeat initial calibration.	Lab Manager/ Analyst	WS-MS-0005 WS-MS-0007 WS-MS-0008 ST-MS-0001 ST-MS-0002

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GC/MS	Retention Time Window Position Establishment	Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	NA	Analyst	WS-MS-0005 WS-MS-0007 WS-MS-0008 ST-MS-0001 ST-MS-0002
GC/MS	Continuing calibration verification (CCV)	Daily, prior to sample analysis and after every 12 hours of analysis time; and at the end of the analytical batch run	All reported analytes and surrogates within ± 20% of true value All reported analytes and surrogated within ± 50% for end of batch CCV	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/ Analyst	WS-MS-0005 WS-MS-0007 WS-MS-0008 ST-MS-0001 ST-MS-0002
GC/MS	Internal Standards	During acquisition of calibration standard	Areas within -50% to +100% of last ICAL mid-point for each CCV	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Lab Manager/ Analyst	WS-MS-0005 WS-MS-0007 WS-MS-0008 ST-MS-0001 ST-MS-0002

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GC-FID	Initial Calibration (ICAL) – minimum five point calibration	Initial calibration prior to sample analysis.	One of the options below: (1) RSD for each analyte ≤ 20%; (2) Linear least squares regression: r ≥ 0.995; (3) non-linear regression: COD (r^2) ≥ 0.99, minimum of 6 points for second order	Evaluate standards, chromatography, and detector response. If problem found with above, correct as appropriate, then repeat initial calibration.	Analyst/ Section Supervisor	DV-GC-0025 ST-CG-0005 ST-GC-0014 ST-GC-0019
GC-FID	Retention Time Window Position Establishment	Once per ICAL and at the beginning of the analytical sequence, for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	NA	Analyst/ Section Supervisor	DV-GC-0025 ST-CG-0005 ST-GC-0014 ST-GC-0019
GC-FID	Initial calibration verification (ICV)	Second-source standard, immediately following ICAL	All project analytes within ± 20% of the expected value from the ICAL	Evaluate data. If problem (e.g., concentrated standard, plugged injector needle) found, correct, then repeat second source verification. If still fails, repeat initial calibration.	Analyst/ Section Supervisor	DV-GC-0025 ST-CG-0005 ST-GC-0014 ST-GC-0019

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GC-FID	Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the sequence with the exception of CCVs for Pesticides multi-component analytes, which are only required before sample analysis	All project analytes within ± 20% of the expected value from the ICAL	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment with written approval from the client or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst/ Section Supervisor	DV-GC-0025 ST-CG-0005 ST-GC-0014 ST-GC-0019

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
ICP-MS	Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months high a high standard at the upper limit of the range	Within + 10% of true value	Dilute samples within the calibration range, or re-establish/verify the LDR	Analyst/ Section Supervisor	WS-MT-0001 ST-MT-0001
ICP-MS	Tuning	Prior to ICAL	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height	Retune instrument and verify	Analyst/ Section Supervisor	WS-MT-0001 ST-MT-0001
ICP-MS	Initial Calibration (ICAL) – minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	3 standards and a blank. Correlation Coefficient of ≥ 0.998	Recalibrate	Analyst/ Section Supervisor	WS-MT-0001 ST-MT-0001
ICP-MS	Second Source Calibration Verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within ± 10% of expected	Recalibrate	Analyst/ Section Supervisor	WS-MT-0001 ST-MT-0001
ICP-MS	Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within ± 10% of expected value	Recalibrate – rerun 10 samples previous to failed CCV.	Analyst/ Section Supervisor	WS-MT-0001 ST-MT-0001

SAP WORKSHEET #24 CONTINUED

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
ICP-MS	Low-level Calibration Chech Standard (Low-level ICV)	Daily	All analytes within + 20% of expected value	Correct problem and repeat ICAL	Analyst/ Section Supervisor	WS-MT-0001 ST-MT-0001
ICP-MS	Interference Check Solutions (ICS)	After ICAL and prior to sample analysis	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD(unless they are a verified trace impurity from one of the spike analytes) ICS-AB: within + 20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples	Analyst/ Section Supervisor	WS-MT-0001 ST-MT-0001

Notes:
CCC = criteria continuing concentration
CCV =continuing calibration verification
COD =coefficient of determination
DCC =daily calibration check
GC =gas chromatograph
IC =ion chromatography
ICAL =initial calibration
ICV = initial calibration verification
MS = mass spectrometer
RF = response factor
RSD = relative standard deviation
SPCC= system performance check compound

SAP WORKSHEET #24 CONTINUED

All analytical instruments will be calibrated and the calibration acceptance criteria met before samples are analyzed. The analytical laboratories will follow calibration procedures that are compliant with the DoD QSM (DoD, 2013) and the published U.S. EPA methods. Calibration standards will be prepared with National Institute for Standards and Testing traceable standards and analyzed per methods requirements.

Worksheet #24 will be used to identify all project-specific analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each.

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SAP WORKSHEET #25 ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICPMS	Clean sample and skimmer cones	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	Intensity of Daily performance check for Rh at least 200000 counts	Check pump tubing, clean lenses as needed.	Test America Chemist	WS-MT-0001
ICPMS	Replace pump windings	Monitor ISTD counts for variation	Instrument performance and sensitivity	As needed	Monitor ISTD counts for variation	Replace windings, recalibrate and reanalyze	Test America Chemist	WS-MT-0001
GC-ECD	Change septum, clean injection port, change or clip column, install new liner	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Test America Chemist	WS-GC-0001
GC-ECD	Change septum, clean injection port, change or clip column, install new liner	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Test America Chemist	WS-GC-0002
GC/MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	TestAmerica Chemist	WS-MS-0007
GC/MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	TestAmerica Chemist	WS-MS-0007
GC/MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	TestAmerica Chemist	WS-MS-0005

SAP WORKSHEET #25 CONTINUED

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	TestAmerica Chemist	WS-MS-0005
Autotitrator	Inspect and clean electrode, clean probe, prime buret, and check rinse water reservoir.	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	TestAmerica Chemist	WS-WC-00028
Ion Chromatograph	Clean micromembrane suppressor and/or replace column as needed.	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	TestAmerica Chemist	WS-WC-0009
Spectrophotomer	Replace Lamp and/or fuse	Sensitivity check	Instrument performance and sensitivity	As needed	ICAL/ICB pass criteria	Recalibrate	TestAmerica Chemist	WS-WC-0020
Spectrophotomer	Wavelength Calibration	Sensitivity check	Instrument performance and sensitivity	Annually	ICAL/ICB pass criteria	Recalibrate	TestAmerica Chemist	WS-WC-0020
CVAA	Replace disposables, flush lines	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Test America Chemist	WS-MT-0005 WS-MT-0007
GC/MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	TestAmerica Chemist	WS-MS-0008

SAP WORKSHEET #25 CONTINUED

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	TestAmerica Chemist	WS-MS-0008
Spectrophotomer	Replace Lamp and/or fuse	Sensitivity check	Instrument performance and sensitivity	As needed	ICAL/ICB pass criteria	Recalibrate	TestAmerica Chemist	WS-WC-0040
Spectrophotomer	Wavelength Calibration	Sensitivity check	Instrument performance and sensitivity	Annually	ICAL/ICB pass criteria	Recalibrate	TestAmerica Chemist	WS-WC-0040
GC/MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	TestAmerica Chemist	WS-MSA-0015
GC/MS	Change septum, clean injection port, change or clip column, install new liner, change trap, leak check autosampler system.	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	TestAmerica Chemist	WS-MSA-0015
Gas Flow Proportional Counter	• Clean instrument • Inspect windows • QA check	• Physical check • Physical check • Background and source count	• Physical check • Physical check • Check deviation	• Daily • High counts and/or background • Daily	• None applicable • No physical defects • Within 3 sigma of 20 day population	• Recalibrate • Instrument maintenance • Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RD-0403

SAP WORKSHEET #25 CONTINUED

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Gamma Spectrometer	• Clean cave; fill dewar with N_2 • QA check	• Physical check • Background and source check	• Physical check • Check deviation	• Weekly • Daily	• Acceptable background • Within 3 sigma of measured population	• Recalibrate • Instrument maintenance Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RD-0102
Alpha Spectrometer	Clean planchette holders	Physical check	Physical check	Monthly	Acceptable background and calibration efficiencies	• Recalibrate • Instrument maintenance • Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RC-0210
Liquid Scintillation Counter	• QA check • Clean dust and debris from sample deck • Photon multiplier tubes cleaned by manufacturer	• Background and efficiency verification for C-14 and H-3 • Physical check • Physical check	• Review of daily control data • Physical check • Physical check	• Daily • Monthly • Semi-annual or annual	For all three maintenance activities: within 3 sigma of established baselines and stable baselines for C-14 and H-3 efficiencies	• Recalibrate • Instrument maintenance • Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RD-0302
Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GCMS GC ICP-MS Cold Vapor AA	Parameter Setup	Physical check	Physical check	Initially; prior to DCC	Predetermined optimum parameter settings	Reset if incorrect	TestAmerica – St. Louis Analyst	ST-MS-0002 ST-MS-0001 ST-GC-0016 ST-GC-0015 ST-GC-0005 ST-GC-0014 ST-GC-0019 ST-MT-0001 ST-MT-0005

SAP WORKSHEET #25 CONTINUED

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Tune Check	Instrument Performance	Conformance to instrument tuning	Initially; prior to DCC	Compliance to ion abundance criteria	Repeat tune check to rule out standard degradation or inaccurate injection. If problem persists, perform retune the instrument and repeat tune check.	TestAmerica – St. Louis Analyst	ST-MS-0002 ST-MS-0001
ICP-MS	ICS	Instrument Performance	Conformance to interference check	Prior to sample analysis	Within + 20% of expected value	Terminate analysis, reanalyze ICS to rule out standard degradation or inaccurate injection. If problem persists, perform instrument maintenance, repeat calibrations and reanalyze all associated samples.	TestAmerica – St. Louis Analyst	ST-MT-0001
ICP-MS, ICP-OES	ICB/CCB	Instrument Performance	Instrument contamination check	After every calibration verification	ICB:No analytes detected > RL; CCB: no analyte detected > 3X MDL	Determine possible source of contamination and apply appropriate measure to correct the problem. Reanalyze calibration blank and all associated samples.	TestAmerica – St. Louis Analyst	ST-MT-0001

SAP WORKSHEET #25 CONTINUED

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Cold Vapor AA	ICB/CCB	Instrument Performance	Instrument contamination check	After every calibration verification	No analytes detected > RL	Determine possible source of contamination and apply appropriate measure to correct the problem. Reanalyze calibration blank and all associated samples.	TestAmerica – St. Louis Analyst	ST-MT-0005
Titration	Visual inspection of glassware used; pipette calibration	Performance	QC check	Day of use	Meet method criteria	Replace; reanalyze any affected samples	TestAmerica – St. Louis Analyst	ST-WC-0012, ST-WC-0019, WT-WC-0036
TRAACS	Check air pressure, change tubing as needed, finish run with wash out	Performance	QC check	Day of use	Meet method criteria	Contact repair service – reanalyze any affected samples.	TestAmerica – St. Louis Analyst	ST-WC-0002
Spectrophotometer	Change membrane as needed	Performance	QC check	Day of use	Meet method criteria	Contact repair service – reanalyze any affected samples.	TestAmerica – St. Louis Analyst	ST-WC-0017
IC	Change guard columns, Replace Tubing, Change Pump Seals	Observation of deteriorating chromatography	Visual Inspection	As needed	No interference/ carry over peaks; no leaks; stable retention times	Replace guard column, replace worn tubing, change pump seals	TestAmerica Analyst	ST-WC-0028
Gamma Spectrometer	• Clean cave; fill dewar with N2 • QA check	• Physical check • Background and source check	• Physical check • Check deviation	• Weekly • Daily	• Acceptable background • Within 3 sigma of measured population	• Recalibrate • Instrument maintenance • Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RD-0102

SAP WORKSHEET #25 CONTINUED

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Alpha Spectrometer	Clean planchette holders	Physical check	Physical check	Monthly	Acceptable background and calibration efficiencies	• Recalibrate • Instrument maintenance • Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RC-0210
Liquid Scintillation Counter	• QA check • Clean dust and debris from sample deck • Photon multiplier tubes cleaned by manufacturer	• Background and efficiency verification for C-14 and H-3 • Physical check • Physical check	• Review of daily control data • Physical check • Physical check	• Daily • Monthly • Semi-annual or annual	For all three maintenance activities: within 3 sigma of established baselines and stable baselines for C-14 and H-3 efficiencies	• Recalibrate • Instrument maintenance • Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RD-0302
Gas Flow Proportional Counter	• Clean instrument • Inspect windows • QA check	• Physical check • Physical check • Background and source count	• Physical check • Physical check • Check deviation	• Daily • High counts and/or background • Daily	• None applicable • No physical defects • Within 3 sigma of 20 day population	• Recalibrate • Instrument maintenance • Consult with Technical Director	TestAmerica – St. Louis Group Leader / Analyst	ST-RD-0403

Notes:

CCB = continuing calibration blank
DCC = daily calibration check
GC = gas chromatograph
IC = ion chromatography
ICB = initial calibration blank
ICS = interference check solution
L/min = liters per minute
MS = mass spectrometer
RL = reporting limit
rps = revolutions per second

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SAP WORKSHEET #26 SAMPLE HANDLING SYSTEM

Sample Handling System
Sample Collection, Packaging, and Shipment
Sample Collection (Personnel/Organization): Field Sampling Team/MMEC Group
Sample Packaging (Personnel/Organization): Field Sampling Team/MMEC Group
Coordination of Shipment (Personnel/Organization): Field Sampling Team/MMEC Group
Type of Shipment/Carrier: Courier and Overnight Shipping
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Chemist/Sample Receiving, TestAmerica
Sample Custody and Storage (Personnel/Organization): Chemist/Sample Receiving, TestAmerica
Sample Preparation (Personnel/Organization): Chemist/Chemist, TestAmerica
Sample Determinative Analysis (Personnel/Organization): Project Chemists/Chemist, TestAmerica
Sample Archiving
Field Sample Storage (Number of days from sample collection): 60 days, or as required on a project specific basis
Sample Extract/Digestate Storage (Number of days from extraction/digestion): 60 days, or as required on a project specific basis
Biological Sample Storage (Number of days from sample collection): Not Applicable
Sample Disposal
Personnel/Organization: Sample Receiving, TestAmerica
Number of Days from Analysis: 30 days, or as required on a project specific basis

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SAP WORKSHEET #27 SAMPLE CUSTODY REQUIREMENTS TABLE

Sample Identification Number

Each sample collected will be assigned a unique sample identification (ID) code used to record and report the results.

The coded sample ID assignments will use these criteria:

- The ID will begin the IR site number displayed as two characters indicating that the sample was collected at that specific IR site, followed by a hyphen.
- The ID of the well the sample was collected from.
- The DATE is six digits identifying the date the sample was collected (YYMMDD).

For example, “07-S7-B32-150825” would be a sample collected from IR Site 7 from monitoring well S7-B32 on August 25, 2015.

QC samples collected during the sampling will use the same coding system as the environmental samples. Field QC designations will conform to the following formats:

Source Blanks: Source blank (SB) sample identifiers will consist of a “SB” label and the sequential number. Example: SB01

Equipment Blanks: Equipment blank (EB) sample identifiers will consist of an “EB” label, and the sequential number. Example: EB01

Trip Blanks: Trip blank (TB) sample identifiers will consist of a “TB” label, and the sequential number. Example: TB01

Temperature Blanks will be labeled as temperature blanks. Temperature blanks are not subject to chemical analysis and the results will not be entered into the database.

Field Documentation

Complete and accurate documentation is essential to ensure proper sample identification and to demonstrate that sampling procedures are carried out as described in the SAP. Field activities and original data generated in the field will be recorded using permanently bound, hardcover, uniquely labeled field logbooks with sequentially numbered pages. The following general guidelines for maintaining field documentation will be followed by project personnel:

- Documentation will be completed in permanent black ink.
- All entries will be legible.
- Errors will be corrected by drawing a single line through the error and writing the correct information; the correction will be initialed and dated.
- Unused pages will be crossed out, and each page will be signed and dated.

In addition, a field logbook will be maintained by the field sampler to summarize chronologically all field activities performed during the course of a given workday. Details on the logbook procedures are discussed in this Worksheet under Field Logbook.

SAP WORKSHEET #27 CONTINUED

All information pertinent to field sampling will be recorded in a field logbook to maintain the integrity and traceability of samples. All samples will be properly labeled and custody sealed before they are transported to the laboratory and will be accompanied by completed chain of custody (COC) documentation. All documentation will be recorded in a field logbook in indelible ink.

No information will be obliterated from a sample label, logbook, instrument calibration form, or COC record. Corrections made to any field forms or logbooks will be made by drawing a line through the error, entering the correct information, and entering initials by the individual making the correction and the date on which the correction was made. If the error is noted after the sample label has been taped for protection or after a custody seal has been affixed to the sample container before being shipped, the field sampler will discard the erroneous label or seal and make a new one. Error corrections made to a sample label or COC form will be noted in the logbook.

Sample Labels

Sample labels serve to prevent misidentification of samples by openly displaying unique identification. Sample containers can be prepared before field work or onsite. Sample labels will be made of weatherproof paper or plastic with a gummed back and will be completed with indelible ink. When necessary, the labels will be covered with clear tape to minimize damage to the label. This information will be entered into the database during sample check in. A description of the sample (including the sample identification number and sample date and time) will be recorded in the field logbook. Any other pertinent information regarding sample identification will be recorded on the sample log sheets or in the field logbooks. The following information will be on each sample label:

- Sample ID code
- Identification of the project site of sample collection
- Name or initials of field sampler
- Analysis required and sample preservation (if applicable)
- Date of sampling (YYYY/MM/DD)
- Local standard time of sample collection, using 24-hour clock notation

Chain of Custody

The field sampler is responsible for creating a COC record where information for each sample collected in the field will be entered. The COC record is necessary to physically trace sample possession from the time of collection to ultimate disposition. Each COC record will be signed as relinquished or received with each change of possession. The following information must be contained in the COC:

- Project name and number
- Names of field samplers
- Sample identification number

SAP WORKSHEET #27 CONTINUED

- Date of sampling
- Local standard time of sample collection, using 24-hour clock notation
- Sample matrix
- Number and type of containers for each sample aliquot
- Type of analysis requested
- Preservation of sample containers (if applicable)
- Means of transmittal to the analytical laboratory or unusual circumstances
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of each individual releasing the shipping container
- Name, address, and individual to receive results

The “COMMENTS/INSTRUCTIONS” line in the COC record will be used to communicate any specific instructions to the analytical laboratory. Additional information relating to a sample may also be noted in the “REMARKS” line. In the event that more than one analytical laboratory will be used, different COC forms will be made for each lab. The number of containers (i.e., coolers) intended to go to a specific analytical laboratory will be made clear on the COC form under the block, “NO. OF COOLERS SHIPPED.” See Attachment 1 for a sample of the COC form.

Field Logbook

A logbook will be maintained by the field sampler to summarize chronologically all field activities performed during the course of a given workday. The logbook is intended to provide interested parties, who are not present in the field at the time of data entry, with all the necessary information about field conditions in order to recreate the event that occurred during field work. Logbooks are to be pre-bound with numbered pages and all entries must be made in indelible ink. To avoid tampering, the user will draw a line across any unused space and initial to signify that no entries were made in those blank pages or spaces by the authorized user. Logbooks will contain, at a minimum, the following information:

- Date of entry and recorder’s name
- Site location
- Sample location, including distances to nearest fixed point (s) of reference
- Sample depth (bgs, if applicable)
- Sample matrix
- Sample appearance
- Volume of sample collected

SAP WORKSHEET #27 CONTINUED

- Field measurements (if applicable)
- Type of sampling equipment used
- Names of all individuals present during sampling
- Sample collection date and times, using 24-hour clock notation
- Sample identification numbers
- Type and number of sample containers used per sampling site
- Designation of QC samples (e.g., blanks, splits, or duplicates)

A logbook is considered a legal document and is admissible as evidence in legal proceedings; therefore, entries made should be factual, detailed, and objective.

Sample Packing and Shipment

Because degradation of samples can occur rapidly following collection because of physical, chemical, and biological factors, it is important to exercise precaution when handling, transporting, or storing samples prior to laboratory analyses. Immediately following collection, the samples will be appropriately labeled, packaged, and placed in a cooler with ice (double bagged in re-sealable bags), to maintain a temperature of between 2 and 6°C. The samples will be delivered to the laboratory at the earliest possible time (but no later than two days following collection) and accompanied by a proper COC record and shipping documentation to track any change in sample possession. Details of the sample packing and shipment procedures are as follows.

Sample packaging and shipment procedures for this project will conform to Department of Transportation/International Air Transport Association procedures as applicable for packaging. All sample containers will be double-bagged using resealable plastic bags to protect the sample from moisture and to minimize the potential for breakage or cross-contamination during transportation to the laboratory. If transported by a commercial carrier, all glass sample containers will be protected with bubble wrap before they are placed in coolers.

Each cooler will be shipped with a temperature blank. The temperature of the cooler will be recorded by the laboratory on the COC record immediately upon receipt of the samples. Sample cooler drain spouts will be taped from the inside and outside of the cooler to prevent leakage.

The samples will be packed in a sample cooler with ice (double bagged in resealable bags) below and above sample containers. Two custody seals will be taped across the cooler lid: one seal in the front and one seal in the back. The COC record will be completed and signed by the courier. The cooler and the top two copies (white and pink) of the COC record will then be released to the courier for transportation to the laboratory.

Saturday deliveries will be coordinated with the laboratory in advance, and field sampling personnel or their designee must confirm that Saturday delivery stickers are placed on each cooler.

SAP WORKSHEET #27 CONTINUED

Laboratory Custody Procedures

Laboratory sample receipt, handling, and custody procedures are provided in more detail in the lab quality systems manual. The lab SOP includes laboratory sample management and COC procedures. At a minimum, the following procedures will be included. The laboratory sample custodian will inspect the integrity of the cooler custody seals and measure the temperature of the samples received using the “Temperature Blank” container included in each cooler. The samples will be checked according to the laboratory “Sample Receiving Checklist” (see laboratory custody SOPs included in Attachment 3) against the COC form for holding times, sample identification, and integrity. The samples will be logged into the laboratory management system. Immediately after receipt, the samples will be stored in an appropriate, secure storage area. Custody of the samples will be maintained and recorded in the laboratory from receipt to analysis and this record will be included with the data package deliverables. If the laboratory sample custodian judges sample custody to be invalid (e.g., samples arrive damaged or custody seals have been broken), the MMEC Group PM will be advised immediately, and the samples will not be analyzed unless the MMEC Group PM so authorizes. The MMEC Group PM, laboratory PMs, and QCM will be notified. The MMEC Group PM will make a decision as to the fate of the sample(s) in question on a case-by-case basis.

The sample(s) will be either processed “as is” with custody failure noted along with the analytical data, or rejected with sampling rescheduled if necessary. Any problem with a sample will be noted in the appropriate data report.

In addition, the lab will follow the laboratory SOPs for proper disposal of the environmental samples in accordance with federal, state, and local ordinances.

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SAP WORKSHEET #28 LABORATORY QC SAMPLES TABLE

Matrix	Water
Analytical Group	ICPMS Metals
Analytical Method/ SOP Reference	U.S. EPA Method 6020/6020A WS-MT-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration blank	After IC, after CCV calibration, after every 10 samples, and at the end of the sequence	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Evaluate blank to determine if instrument or solution caused, then correct. Re-prepare and reanalyze the blank. All samples following the last acceptable calibration blank must be reanalyzed.	Lab Manager/ Analyst	Accuracy	No target analytes ≥ ½ RL.
Method blank	One per digestion batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with DoD QSM requirements.*	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Lab Manager/ Analyst	Accuracy/Bias Contamination	No target analytes ≥ ½ RL in accordance with DoD QSM requirements

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	ICPMS Metals
Analytical Method/ SOP Reference	U.S. EPA Method 6020/6020A WS-MT-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One LCS per each preparation batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager/ Analyst	Precisions and Accuracy/Bias	QC acceptance criteria: as specified by DoD; or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	ICPMS Metals
Analytical Method/ SOP Reference	U.S. EPA Method 6020/6020A WS-MT-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD for all analytes	One MS/MSD pair per preparation batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 20% (between MS and MSD).	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Lab Manager/ Analyst	Precision and Accuracy/Bias	QC acceptance criteria: as specified by DoD (RPD ≤ 20); or laboratory statistically derived control limits
Dilution test	Each new sample matrix	1:5 dilution must agree within ±10% of the original determination for concentrations > 50x limit of quantitation (LOQ) prior to dilution.	Perform post-digestion spike addition in accordance with DoD QSM requirements.	Lab Manager/ Analyst	Accuracy	10% Difference
Post digestion spike addition	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 80% to 120% of expected results for compounds < 50x LOQ in native sample.	Flag in accordance with DoD QSM requirements.	Lab Manager/ Analyst	Accuracy	Recovery within 80% to 120% of expected results in accordance with DoD QSM requirements

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	ICPMS Metals
Analytical Method/ SOP Reference	U.S. EPA Method 6020/6020A WS-MT-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards (IS)	Every sample.	IS intensity within 30-120% of the IS in the ICAL blank	Reanalyze sample at a 5-fold dilution with the addition of appropriate amounts of internal standards. For failed QC samples, correct problem, and rerun all associated failed field samples.	Lab Manager/ Analyst	Accuracy/Bias	IS outside limits is an indicator of matrix effects.

Notes:

* Common laboratory contaminants are Al, Ca, Fe, K, Mg, Na, Pb, Sn, and Zn.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Pesticides
Analytical Method/ SOP Reference	U.S. EPA Method 8081B WS-GC-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with DoD QSM requirements.	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ ½ RL in accordance with DoD QSM requirements

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Pesticides
Analytical Method/ SOP Reference	U.S. EPA Method 8081B WS-GC-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One LCS per preparation batch, spiked with single-eluting pesticides only.	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria: as specified by DoD; or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Pesticides
Analytical Method/ SOP Reference	U.S. EPA Method 8081B WS-GC-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate Standards	Each sample	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria: as specified by DoD; or laboratory statistically derived control limits
MS/MSD for all analytes	One MS/MSD pair per preparation batch, spiked with single-eluting pesticides only.	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 30% (between MS and MSD).	If not related to matrix interference, re-extract and reanalyze MS/MSD.	Lab Manager / Analyst	Precision and Accuracy/Bias	QC acceptance criteria: as specified by DoD; or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Pesticides
Analytical Method/ SOP Reference	U.S. EPA Method 8081B WS-GC-0001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Confirmation of positive results (second column & detector)	All positive results must be confirmed	Calibration and QC criteria are the same as for initial or primary column analysis. RPD of results between primary and secondary column analysis ≤ 40%.	Evaluate data, then report with flag to denote RPD > 40%. Narrate obvious matrix issues.	Lab Manager / Analyst	Precision and Accuracy	Results with high RPD qualified as per DoD QSM.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PCBs
Analytical Method/ SOP Reference	U.S. EPA Method 8082 WS-GC-0002

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with DoD QSM requirements.	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ ½ RL in accordance with DoD QSM requirements

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PCBs
Analytical Method/ SOP Reference	U.S. EPA Method 8082 WS-GC-0002

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One LCS per preparation batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria: as specified by DoD; or laboratory statistically derived control limits
MS/MSD for all analytes	One MS/MSD pair per preparation batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 30% (between MS and MSD).	If not related to matrix interference, re-extract and reanalyze MS/MSD.	Lab Manager / Analyst	Precision and Accuracy/Bias	QC acceptance criteria: as specified by DoD; or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PCBs
Analytical Method/ SOP Reference	U.S. EPA Method 8082 WS-GC-0002

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate standards	All field and QC samples.	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	Accuracy/Bias	QSM or laboratory statistically derived control limits
Confirmation of positive results (second column & detector)	All positive results must be confirmed	Calibration and QC criteria are the same as for initial or primary column analysis. RPD of results between primary and secondary column analysis ≤ 40%.	Evaluate data, then report with flag to denote RPD > 40%. Narrate obvious matrix issues.	Lab Manager / Analyst	Precision and Accuracy	Results with high RPD qualified as per DoD QSM.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Volatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8260B / WS-MS-0007

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
Internal standards	During acquisition of calibration standard, samples, and QC check samples	Retention time within ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Lab Manager / Analyst	Meets all U.S. EPA Method requirements
Method blank	One per batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants as noted in Table 15, no analytes detected >RL in accordance with DoD QSM requirements	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Lab Manager / Analyst	No target analytes ≥ RL

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Volatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8260B / WS-MS-0007

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
MS/MSD	One MS/MSD per batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 20% (between MS and MSD).	If not related to matrix interference, re-extract and reanalyze MS/MSD.	Lab Manager / Analyst	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Volatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8260B / WS-MS-0007

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
LCS	One LCS per batch	QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Volatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8260B / WS-MS-0007

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
Surrogate standards	All field and QC samples.	QSM limits (if available) or current in-house limits if no QSM limits published.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Semivolatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
Internal standards	During acquisition of calibration standard, samples, and QC check samples	Retention time within ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Lab Manager / Analyst	Meets all U.S. EPA Method requirements
Method blank	One per batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants as noted in Table 15, no analytes detected >RL in accordance with DoD QSM requirements	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Lab Manager / Analyst	No target analytes ≥ RL

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Semivolatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
MS/MSD	One MS/MSD per batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 20% (between MS and MSD).	If not related to matrix interference, re-extract and reanalyze MS/MSD.	Lab Manager / Analyst	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Semivolatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
LCS	One LCS per batch	QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Semivolatiles
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Project Specific Measurement Performance Criteria
Surrogate standards	All field and QC samples.	QSM limits (if available) or current in-house limits if no QSM limits published.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Alkalinity
Analytical Method/ SOP Reference	SM 2320B WS-WC-0028

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per batch	No target analytes ≥ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank.	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ RL.
LCS (Evaluated only for total alkalinity)	One LCS per preparation batch	Recovery 90-110%	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Accuracy/Bias	QC acceptance criteria.
Sample Duplicate	One duplicate per analysis batch	RPD ≤ 20% between sample and duplicate.	Evaluate instrument and reanalyze once. Narrate if remains out of control.	Lab Manager / Analyst	Precision	QC acceptance criteria.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Anions by IC
Analytical Method/ SOP Reference	U.S. EPA 300 WS-WC-0009

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ ½ RL in accordance with DoD QSM requirements
LCS	One LCS per analytical/preparation batch, spiked with all analytes to be reported.	QSM limits (if available), otherwise recovery 90-110% for aqueous samples; 85-115% for solid samples.	Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Anions by IC
Analytical Method/ SOP Reference	U.S. EPA 300 WS-WC-0009

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD for all analytes	One MS/MSD pair per preparation batch	QSM limits (if available), otherwise recover 90-110% for aqueous samples; 85-115% for solid samples. RPD ≤ 15% between MS and MSD.	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Lab Manager / Analyst	Precision and Accuracy/Bias	QC acceptance criteria.

SAP WORKSHEET #28 CONTINUED

Matrix	Water and Solid
Analytical Group	Chromium VI
Analytical Method/ SOP Reference	U.S. EPA 7196A WS-WC-0020

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Evaluate calibration blanks. If reagent issue, prepare fresh reagents, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank.	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ ½ RL in accordance with DoD QSM requirements.
LCS	One LCS per preparation batch	QSM limits (if available) or current in-house limits if no QSM limits published	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch, if sufficient sample material is available.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QSM control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water and Solid
Analytical Group	Chromium VI
Analytical Method/ SOP Reference	U.S. EPA 7196A WS-WC-0020

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD for all analytes	One MS/MSD pair per preparation batch (Aqueous samples, one per every 10 project samples per matrix, Solid, one pair per preparatory batch per matrix)	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 20% (between MS and MSD).	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Lab Manager / Analyst	Precision and Accuracy/Bias	QSM control limits

Notes:

Laboratory does not perform Method 3060 digestion on solids; therefore, pre- and post-digestion spikes are not included in this table.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Mercury
Analytical Method/ SOP Reference	U.S. EPA Method 7470A/7471A WS-MT-0005 / WS-MT-0007

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration blank	After IC, after CCV calibration, after every 10 samples, and at the end of the sequence	No target analytes > LOD in accordance with DoD QSM requirements	Evaluate blank to determine if instrument or solution caused, then correct. Re-prepare and reanalyze the blank. All samples following the last acceptable calibration blank must be reanalyzed.	Lab Manager / Analyst	Accuracy	No target analytes > LOD in accordance with DoD QSM requirements
Method blank	One per digestion batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ ½ RL in accordance with DoD QSM requirements
MS/MSD	One MS/MSD pair per preparation batch	QC acceptance criteria as specified by DoD.	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria as specified by DOD.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Mercury
Analytical Method/ SOP Reference	U.S. EPA Method 7470A/7471A WS-MT-0005 / WS-MT-0007

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One LCS per each preparation batch	QC acceptance criteria as specified by DoD.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria as specified by DOD.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PAHs
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0008

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards	During acquisition of calibration standard, samples, and QC check samples	Retention time within ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Lab Manager / Analyst	Precisions and Accuracy/Bias	Meets all U.S. EPA Method requirements
Method blank	One per analytical batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with DoD QSM requirements	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ RL

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PAHs
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0008

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	One MS/MSD per analytical/preparation batch	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 20% (between MS and MSD).	If not related to matrix interference, re-extract and reanalyze MS/MSD.	Lab Manager / Analyst	Precision and Accuracy/Bias	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PAHs
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0008

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One LCS per analytical/preparation batch	QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precision and Accuracy/Bias	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PAHs
Analytical Method/ SOP Reference	U.S. EPA Method 8270D / WS-MS-0008

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate standards	All field and QC samples.	QSM limits (if available) or current in-house limits if no QSM limits published.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, re-extract and reanalyze.	Lab Manager / Analyst	Accuracy/Bias	QSM or laboratory statistically derived control limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	COD
Analytical Method/ SOP Reference	U.S. EPA 410.4 WS-WC-0040

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per batch	COD < RL	Correct problem, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank.	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ RL.
LCS	One LCS per preparation batch	Recovery 85-115%.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria. 85 - 115

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	COD
Analytical Method/ SOP Reference	U.S. EPA 410.4 WS-WC-0040

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD for all analytes	One MS/MSD pair per preparation batch	Recovery 75-125% RPD ≤ 20% between MS and MSD.	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Lab Manager / Analyst	Precision and Accuracy/Bias	QC acceptance criteria. 75-125

SAP WORKSHEET #28 CONTINUED

Matrix	Water/Soil
Analytical Group	Gross Alpha/Beta
Analytical Method/SOP Reference	U.S. EPA 900.0 MOD/SOP ST-RD-0403

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – St. Louis Analyst	Accuracy	Analytes < RL
LCS and/or LCD	1 per preparatory batch	Recovery: Gross Alpha: 73–133% Gross Beta: 75–125% RPD ≤40% and/or RER ≤1	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
MS	As requested	Recovery: Gross Alpha: 35–150% Gross Beta: 89–143% RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the MS	TestAmerica – St. Louis Analyst	Accuracy/Precision	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water/Soil
Analytical Group	Gross Alpha/Beta
Analytical Method/SOP Reference	U.S. EPA 900.0 MOD/SOP ST-RD-0403

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – St. Louis Analyst	Accuracy	Analytes < RL
LCS	1 per preparatory batch	Recovery Limits: Cs-137: 90-110% Co-60: 89-110% Am-241: 90-110%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Air
Analytical Group	Volatiles
Analytical Method/ SOP Reference	U.S. EPA Method TO-15 / WS-MSA-0015

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Check of mass spectral ion intensities (tuning procedure) using BFB (TO-15)	Prior to initial calibration and calibration verification	Must meet the method requirements before samples are analyzed in accordance with DoD QSM requirements	Retune instrument and verify the tune acceptability in accordance with DoD QSM requirements	Lab Manager / Analyst	Sensitivity	Meets all U.S. EPA Method requirements
Internal standards	During acquisition of calibration standard, samples, and QC check samples	Areas within 60% to 140% of midpoint of the last ICAL for each sample and QC.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning. If field samples still outside criteria, qualify data and explain in case narrative.	Lab Manager / Analyst	Precisions and Accuracy/Bias	Meets all U.S. EPA Method requirements

SAP WORKSHEET #28 CONTINUED

Matrix	Air
Analytical Group	Volatiles
Analytical Method/ SOP Reference	U.S. EPA Method TO-15 / WS-MSA-0015

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per analytical batch	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with DoD QSM requirements	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ 1/2 RL

SAP WORKSHEET #28 CONTINUED

Matrix	Air
Analytical Group	Volatiles
Analytical Method/ SOP Reference	U.S. EPA Method TO-15 / WS-MSA-0015

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One LCS per analytical/preparation batch	QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Precision and Accuracy/Bias	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 43 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	VOCs
Analytical Method	Method 8260C

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards	Every field sample and QC samples	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Correct problem, then re-reanalyze affected samples	Lab Manager/ Analyst	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 24 and Appendix B Table 4	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 24 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	VOCs
Analytical Method	Method 8260C

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 24 and Appendix B Table 4
Surrogate standards	Every field sample and QC sample	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 24 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	VOCs
Analytical Method	Method 8260C SIM

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards	Every field sample and QC samples	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Correct problem, then re-reanalyze affected samples	Lab Manager/ Analyst	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD per preparation batch per matrix	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 30% (between MS and MSD).	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 24 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	VOCs
Analytical Method	Method 8260C SIM

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 30%	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 24 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	SVOCs
Analytical Method	EPA Method 8270D

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards	Every field sample and QC samples	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Correct problem, then re-reanalyze affected samples	Lab Manager/ Analyst	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD of all analytes ≤ 30% (between MS and MSD).	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	SVOCs
Analytical Method	EPA Method 8270D

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4
Surrogate standards	Every field sample and QC sample	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published.	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PAHs
Analytical Method	EPA Method 8270D SIM

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards	Every field sample and QC samples	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Correct problem, then re-reanalyze affected samples	Lab Manager/ Analyst	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 28 and Appendix B Table 4	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 28 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PAHs
Analytical Method	EPA Method 8270D SIM

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 28 and Appendix B Table 4	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 28 and Appendix B Table 4
Surrogate standards	Every field sample and QC sample	Percent Recoveries per DoD QSM 5.0 Appendix C Table 28 and Appendix B Table 4	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 28 and Appendix B Table 4

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Pesticides
Analytical Method	EPA Method 8081B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Pesticides
Analytical Method	EPA Method 8081B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1
Surrogate standards	Every field sample and QC sample	Percent Recoveries per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Pesticides
Analytical Method	EPA Method 8081B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Second column confirmation of positive results	All positive results must be confirmed	Calibration and QC criteria for second column are the same as for initial and primary column analysis. Results between primary and secondary column RPD ≤ 40%.	N/A	Lab Manager/ Analyst	Precision	RPD of results between two columns ≤ 40%.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PCBs
Analytical Method	EPA Method 8082A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 17 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 17 and Appendix B Table 1
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 17 and Appendix B Table 1	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 17 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	PCBs
Analytical Method	EPA Method 8082A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes			
Surrogate standards	Every field sample and QC sample	Percent Recoveries per DoD QSM 5.0 Appendix C Table 17 and Appendix B Table 1	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 17 and Appendix B Table 1
Second column confirmation of positive results	All positive results must be confirmed	Calibration and QC criteria for second column are the same as for initial and primary column analysis. Results between primary and secondary column RPD ≤ 40%.	N/A	Lab Manager/ Analyst	Precision	RPD of results between two columns ≤ 40%.

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	TPH-DRO/MRO
Analytical Method	EPA Method 8015B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	TPH-DRO/MRO
Analytical Method	EPA Method 8015B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1
Surrogate standards	Every field sample and QC sample	Percent Recoveries per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	TPH-GRO
Analytical Method	EPA Method 8015B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	TPH-GRO
Analytical Method	EPA Method 8015B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 14 and Appendix B Table 1
Surrogate standards	Every field sample and QC sample	Percent Recoveries of 69-150% (lab-derived statistical limits)	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries of 69-150% (lab-derived statistical limits)

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Dissolved Gases
Analytical Method	RSK-175

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 42 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 42 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Dissolved Gases
Analytical Method	RSK-175

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 42 and Appendix B Table 1	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 42 and Appendix B Table 1

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Metals
Analytical Method	EPA Method 6020A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 6 and Appendix B Table 9	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 6 and Appendix B Table 9

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Metals
Analytical Method	EPA Method 6020A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 6 and Appendix B Table 9	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 6 and Appendix B Table 9

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Metals
Analytical Method	EPA Method 6020A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial and Continuing Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 × MDL	Any sample associated with a blank that fails the criteria checks will be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results will be reported with appropriate data qualifying codes.	TestAmerica - St. Louis Analyst	Accuracy	No analytes detected > 2 × MDL
Serial dilution	Each new sample matrix	1:5 dilution must agree within ±10% of original determination.	Perform post-digestion spike if serial dilution does not meet criteria	TestAmerica - St. Louis Analyst	Accuracy	1:5 dilution must agree within ±10% of original determination.
Post-digestion spike	When serial dilution or matrix spike fails	Recovery within 80-120%	Re-analyze post-digestion spike.	TestAmerica - St. Louis Analyst	Accuracy	Recovery within 80-120%

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Mercury
Analytical Method	EPA Method 7470A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Mercury
Analytical Method	EPA Method 7470A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Mercury
Analytical Method	EPA Method 7470A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 × MDL	Any sample associated with a blank that fails the criteria checks will be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results will be reported with appropriate data qualifying codes.	TestAmerica - St. Louis Analyst	Accuracy	No analytes detected > 2 × MDL

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Total Cyanide
Analytical Method	EPA Method 9012B

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
LCS	One LCS per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 39 and Appendix B Table 11	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 39 and Appendix B Table 11

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Total Cyanide
Analytical Method	EPA Method 9012B

QC Sample	**Frequency / Number**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
MS	One MS per preparation batch per matrix	Percent Recovery per DoD QSM 5.0 Appendix C Table 39 and Appendix B Table 11	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recovery per DoD QSM 5.0 Appendix C Table 39 and Appendix B Table 11
Duplicate	One DUP per preparation batch per matrix	RPD per DoD QSM 5.0 Appendix B Table 11	Correct problem, then re-reanalyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD per DoD QSM 5.0 Appendix B Table 11

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Total Sulfide
Analytical Method	EPA Method 376.1

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results
LCS	One LCS per preparation batch per matrix	Percent Recoveries: 90-110% (lab-derived statistical limits)	Correct problem, then re-reanalyze the LCS and all associated batch samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries: 90-110% (lab-derived statistical limits)
MS	One MS per preparation batch per matrix	Percent Recoveries: 90-110% (lab-derived statistical limits)	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries: 90-110% (lab-derived statistical limits)
Duplicate	One DUP per preparation batch per matrix	RPD Limit: 20% (lab-derived statistical limits)	Correct problem, then re-reanalyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD Limit: 20% (lab-derived statistical limits)

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Hexavalent Chromium
Analytical Method	EPA Method 7196A

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
LCS	One LCS per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 10 and Appendix B Table 10	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 10 and Appendix B Table 10

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Hexavalent Chromium
Analytical Method	EPA Method 7196A

QC Sample	**Frequency / Number**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
MS	One MS per preparation batch per matrix	Percent Recovery per DoD QSM 5.0 Appendix C Table 10 and Appendix B Table 10	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recovery per DoD QSM 5.0 Appendix C Table 10 and Appendix B Table 10
Duplicate	One DUP per preparation batch per matrix	RPD per DoD QSM 5.0 Appendix B Table 10	Correct problem, then re-reanalyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD per DoD QSM 5.0 Appendix B Table 10

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Anions
Analytical Method	EPA Method 300.0

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
LCS	One LCS per preparation batch per matrix	Percent Recoveries: 90-110% for each analyte	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Percent Recoveries: 90-110% for each analyte

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Anions
Analytical Method	EPA Method 300.0

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS	One MS per preparation batch per matrix	Percent Recoveries: 90-110% for each analyte	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries: 90-110% for each analyte
Duplicate	One DUP per preparation batch per matrix	RPD: 20% for each analyte	Correct problem, then re-reanalyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD: 20% for each analyte

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	TDS
Analytical Method	EPA Method 160.1

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
LCS	One LCS per preparation batch per matrix	Percent Recoveries: 90-110%	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Percent Recoveries: 90-110%

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	TDS
Analytical Method	EPA Method 160.1

QC Sample	**Frequency / Number**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate	One DUP per preparation batch per matrix	RPD: 20%	Correct problem, then re-reanalyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD: 20%

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Alkalinity
Analytical Method	EPA Method 310.1

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
LCS	One LCS per preparation batch per matrix	Percent Recoveries: 90-110%	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Percent Recoveries: 90-110%

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Alkalinity
Analytical Method	EPA Method 310.1

QC Sample	**Frequency / Number**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
MS	One MS per preparation batch per matrix	Percent Recoveries: 80-120%	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries: 80-120%
Duplicate	One DUP per preparation batch per matrix	RPD: 20% for each analyte	Correct problem, then re-reanalyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD: 20% for each analyte

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Gross Alpha/Beta
Analytical Method	EPA Method 900.0

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – -St. Louis Analyst	Accuracy	Analytes < RL
LCS and/or LCD	1 per preparatory batch	Recovery: Gross Alpha: 73–133% Gross Beta: 75–125% RPD ≤40% and/or RER ≤1	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
MS	As requested	Recovery: Gross Alpha: 35–150% Gross Beta: 89–143%	Correct problem, then re-reanalyze all samples processed with the MS	TestAmerica – St. Louis Analyst	Accuracy/ Precision	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Gamma Spec
Analytical Method	EPA Method 901.1

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – St. Louis Analyst	Accuracy	Analytes < RL
LCS	1 per preparatory batch	Recovery Limits: Cs-137: 90-111% Co-60: 89-110% Am-241: 90-111%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Radium-226
Analytical Method	EPA Method 903.0

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – St. Louis Analyst	Accuracy	Analytes < RL
LCS	1 per preparatory batch	Recovery Limits: 68-137%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Radium-228
Analytical Method	EPA Method 904.0

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – St. Louis Analyst	Accuracy	Analytes < RL
LCS	1 per preparatory batch	Recovery Limits: 56-140%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Sr-90
Analytical Method	EPA Method 905.0

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – St. Louis Analyst	Accuracy	Analytes < RL
LCS	1 per preparatory batch	Recovery Limits: 90-134%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Tritium
Analytical Method	EPA Method 906.0

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – -St. Louis Analyst	Accuracy	Analytes < RL
LCS	1 per preparatory batch	Percent Recovery: 74–114%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
MS	As requested	Percent Recovery: 67–130%	Correct problem, then re-reanalyze all samples processed with the MS	TestAmerica – St. Louis Analyst	Accuracy/ Precision	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Isotopic Uranium
Analytical Method	HASL 300 A-01-R

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per preparatory batch	Analytes < RL	Correct problem, then re-reanalyze method blank and all samples processed with the contaminated blank	TestAmerica – St. Louis Analyst	Accuracy	Analytes < RL
LCS	1 per preparatory batch	Percent Recovery: U-234: 84–120% U-238: 83-121%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

SAP WORKSHEET #28 CONTINUED

Matrix	Water
Analytical Group	Isotopic Uranium
Analytical Method	HASL 300 A-01-R

QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Tracer	Per sample, blank, LCS, MS, MSD	U-232 tracer: ≥30% and ≤110%	Truncate carriers/tracers above 100% recovery to eliminate low biased results. Reprep and reanalyze sample if carrier is low (indicating high biased results) if there is activity in the sample above the reporting limit. No reanalysis if matrix interference is nonconformance during sample preparation.	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits
Sample Duplicate	1 per preparatory batch	RPD ≤40% and/or RER ≤1	Correct problem, then re-reanalyze all samples processed with the duplicate	TestAmerica – St. Louis Analyst	Accuracy	Within in-house limits

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SAP WORKSHEET #29 PROJECT DOCUMENTS AND RECORDS TABLE

Document	Where Maintained
Work Plan	MMEC Group Project File and NAVFAC SW Administrative Record
Sampling and Analysis Plan	MMEC Group Project File and NAVFAC SW Administrative Record
Accident Prevention Plan and Site Safety and Health Plan	MMEC Group Project File and NAVFAC SW Administrative Record
Field notes/logbook	MMEC Group Project File
Chain of custody forms	MMEC Group Project File and NAVFAC SW Administrative Record
Audit checklists/reports	MMEC Group Project File
Field photographs	MMEC Group Project File
Laboratory analytical reports including the data packages (Level III and Level IV) as follows: • Table of contents • Signed cover letter • Chain of custody • Sample receipt form 1 • Communication logs (e-mails, phone logs etc.) • Data qualifier table • Case narrative • Results summary forms • QC summary forms • Calibration summary forms • Extraction/analysis logs • Nonconformance report (if applicable) • Raw data for samples, associated laboratory QC samples and calibrations	MMEC Group, Laboratory Project File, and NAVFAC SW Administrative Record
Third-party Level III and Level IV data validation reports	MMEC Group Project File and NAVFAC SW Administrative Record
Validated Laboratory Electronic Data Package	MMEC Group Project File and NAVFAC NEDD NIRIS Website

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SAP WORKSHEET #30 ANALYTICAL SERVICES TABLE

For this project, analytical services will be provided by TBD. Turnaround times for the laboratory data package will be based on the date on which the laboratory receives the samples. Preliminary results from TBD will be sent electronically via e-mail or web portal within 7 working days, and the final data package will be sent in electronic and hardcopy formats to the MMEC Group office within 10 working days. The backup laboratory for this project has yet to be determined. Both labs will be currently certified by the California Department of Health Services Environmental Laboratory Accreditation Program (ELAP), and will have received accreditation from a Department of Defense ELAP-accrediting body for analysis of hazardous materials for the methods specified in this SAP. Copies of the laboratory certifications are presented in Attachment 2. Status of laboratory certifications will be verified during project preparation and before samples are sent to the laboratory. If the laboratory's certification expires during the project execution, a copy of the renewed certification will be obtained to ensure that the laboratory is qualified to perform the analysis.

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization
Water	TPH	See Worksheet #20	U.S. EPA 8015M	Standard	Test America	Curtis & Tompkins
Water	VOCs	See Worksheet #20	U.S. EPA 8260B	Standard	TestAmerica	Curtis & Tompkins
Water	1-, 4-Dioxane	See Worksheet #20	U.S. EPA 8270 SIM	Standard	TestAmerica	Curtis & Tompkins
Water	SVOCs	See Worksheet #20	U.S. EPA 8270C	Standard	TestAmerica	Curtis & Tompkins
Water	PAHs	See Worksheet #20	U.S. EPA 8270C SIM	Standard	TestAmerica	Curtis & Tompkins
Water	Pesticides	See Worksheet #20	U.S. EPA 8081A	Standard	TestAmerica	Curtis & Tompkins
Water	PCBs	See Worksheet #20	U.S. EPA 8082	Standard	TestAmerica	Curtis & Tompkins
Water	Dissolved Metals	See Worksheet #20	U.S. EPA 6010B/6020A/7470A/7196A	Standard	TestAmerica	Curtis & Tompkins
Water	Cyanide	See Worksheet #20	U.S. EPA 335.2	Standard	TestAmerica	Curtis & Tompkins
Water	Dissolved Gasses	See Worksheet #20	RSK175	Standard	TestAmerica	Curtis & Tompkins

SAP WORKSHEET #30 CONTINUED

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization
Water	Anions	See Worksheet #20	U.S. EPA 300	Standard	TestAmerica	Curtis & Tompkins
Water	Alkalinity	See Worksheet #20	U.S. EPA 310.2	Standard	TestAmerica	Curtis & Tompkins
Water	Sulfide	See Worksheet #20	U.S. EPA 376.2	Standard	TestAmerica	Curtis & Tompkins
Water	TDS	See Worksheet #20	U.S. EPA 150.1	Standard	TestAmerica	Curtis & Tompkins
Water	BOD	See Worksheet #20	U.S. EPA 405.1	Standard	TestAmerica	Curtis & Tompkins
Water	COD	See Worksheet #20	U.S. EPA 410.4	Standard	TestAmerica	Curtis & Tompkins
Water	TOC	See Worksheet #20	U.S. EPA 415.1	Standard	TestAmerica	Curtis & Tompkins
Water	DOC	See Worksheet #20	U.S. EPA 5310C	Standard	TestAmerica	Curtis & Tompkins
Water	Census DNA	See Worksheet #20	qPCR	Standard	TBD	TBD
Water	Gross Alpha/Gross Beta	See Worksheet #20	U.S. EPA 9310/900.0	Standard	TestAmerica	Curtis & Tompkins
Water	Radium-226	See Worksheet #20	U.S. EPA 9315/903.0	Standard	TestAmerica	Curtis & Tompkins
Water	Tritium	See Worksheet #20	U.S. EPA 906.0	Standard	TestAmerica	Curtis & Tompkins
Water	Total Radium	See Worksheet #20	U.S. EPA 904.0	Standard	TestAmerica	Curtis & Tompkins
Water	Strontium-90	See Worksheet #20	U.S. EPA 905.0	Standard	TestAmerica	Curtis & Tompkins

SAP WORKSHEET #30 CONTINUED

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization
Water	Gamma Emitting Radionuclides	See Worksheet #20	U.S. EPA 901.1	Standard	TestAmerica	Curtis & Tompkins
Water	Uranium Isotopes	See Worksheet #20	U.S. EPA 908 Modified	Standard	TestAmerica	Curtis & Tompkins
Soil Gas	Fixed Gasses	See Worksheet #20	U.S. EPA TO-03	Standard	TestAmerica	Curtis & Tompkins
Soil Gas	VOCs	See Worksheet #20	U.S. EPA TO-15	Standard	TestAmerica	Curtis & Tompkins

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SAP WORKSHEET #31 PLANNED PROJECT ASSESSMENTS TABLE

Assessment Type[1]	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions	Person(s) Responsible for Monitoring Effectiveness of Corrective Actions
Operational Readiness Review	Prior to sampling activity	Internal	MMEC Group	PM, MMEC Group	PM, MMEC Group	PM, MMEC Group	PM, MMEC Group
Field Sampling Surveillance	During sampling activity	Internal	MMEC Group	QCM, MMEC Group	PM, MMEC Group	QCM, MMEC Group	QCM, MMEC Group MMEC Group PM, MMEC Group
Field Documentation Review	Daily	Internal	MMEC Group	QCM, MMEC Group PM, MMEC Group	PM, MMEC Group	QCM, MMEC Group	QCM, MMEC Group PM, MMEC Group
Data Review Surveillance	Once	Internal	MMEC Group	QCM, MMEC Group	PM, MMEC Group	QCM, MMEC Group	QCM, MMEC Group PM, MMEC Group

Notes:

1. Attachment 1 includes a copy of the sample field audit and review forms.

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SAP WORKSHEET #32 ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

Assessment Type[1]	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Timeframe for Response
Operational Readiness Review	Audit Report	Matt Brookshire MMEC Group	1 day after completion of the inspection	Corrective Action Report	Matt Brookshire, QCM, MMEC Group	5 days
Field Sampling Surveillance	Audit Report	Matt Brookshire MMEC Group	1 day after completion of the inspection	Corrective Action Report	Matt Brookshire, QCM, MMEC Group	5 days
Field Documentation Review	Audit Report	Matt Brookshire MMEC Group	1 day after completion of the inspection	Corrective Action Report	Matt Brookshire, QCM, MMEC Group	5 days
Data Review Surveillance	Audit Report	Matt Brookshire MMEC Group	2 days after completion of the inspection	Corrective Action Report	Matt Brookshire, QCM, MMEC Group	5 days

Notes:

1. Attachment 1 includes a copy of the sample field audit and review forms.

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SAP WORKSHEET #33 QA MANAGEMENT REPORTS TABLE

Type of Report[1]	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Operational Readiness Review	Prior to initiating field work and periodically throughout the duration of the project.	5 days after completion	Matt Brookshire, QCM, MMEC Group	MMEC Group PM
Field Sampling Surveillance	After all data have been generated and reviewed.	5 days after completion	Matt Brookshire, QCM, MMEC Group	MMEC Group PM
Field Documentation Review	After all data have been generated and reviewed.	5 days after completion	Matt Brookshire, QCM, MMEC Group	MMEC Group PM
Data Review Surveillance	After all data have been generated and reviewed.	5 days after completion	Matt Brookshire, QCM, MMEC Group	MMEC Group PM
Corrective Action Plan	After all data have been generated and reviewed.	Preliminary Draft of report approximately 60 days after validated data received. Final report delivery to be determined after internal and regulatory review cycles have been established.	Matt Brookshire, PM, MMEC Group	NAVFAC SW RPMs, Water Board

Notes:

1. Attachment 1 includes a copy of the sample field audit and review forms.

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SAP WORKSHEET #34 VERIFICATION (STEP I) PROCESS TABLE

Verification Input	Description	Internal/External	Responsible for Verification
Chain of Custody Forms	Content of the COC form will be reviewed daily and verified for completeness against the samples within the associated cooler. A copy of the COC form will be filed with other project documents in the assigned project file. The original COC form will be taped on the inside of the cooler for sample shipment.	Internal	Kevin Olness, MMEC Group
Field Notes/ Logbook	Field sampling data, i.e., field logbooks and field forms, will be reviewed and verified for completeness. Field notes/logbook will be forwarded to the PM and placed in the project file.	Internal	Kevin Olness, MMEC Group
Audit Reports	Upon report completion, a copy of all audit reports will be placed in the project file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the project file.	Internal	Matt Brookshire, MMEC Group
Analytical Data Package	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be verified externally and internally according to the data validation process specified in Worksheet #36 of this SAP.	Internal/ External	Lab - TestAmerica Linda Rauto, LDC
Electronic Data Deliverables	All electronic data deliverables will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received electronic data deliverables will be verified externally and internally against the hard copy laboratory data packages	Internal/ External	Lab - TestAmerica Linda Rauto, LDC

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SAP WORKSHEET #35 VALIDATION (STEPS IIA AND IIB) PROCESS TABLE

Step IIa/IIb[1]	Validation Input	Description	Responsible for Validation
IIa	Communication	Establish that the required communication procedures were followed by field or laboratory personnel.	Matt Brookshire, MMEC Group
IIa	Methods (sampling and analysis)	Establish that required sampling and analytical methods were implemented and that any deviations were noted. Evaluate whether proper procedures met performance criteria.	TestAmerica
IIa	Location, maps, and sample ID numbers	Verify the accuracy and precision of the information under scrutiny.	Matt Brookshire, MMEC Group
IIa	Holding times	Ensure that samples were analyzed within holding times specified in method, procedure, or contract requirements. If holding times were not met, confirm that deviations were documented and appropriate notifications were made.	TestAmerica
IIa	List of project-specific analytes	Establish that the project-specific analytes were reported as specified in governing documents (i.e., analytical method, contract, etc.).	TestAmerica
IIa	Field logbook	On a weekly basis, the QCM or assignee will review and verify completeness of the information in the field logbooks as described in Worksheet #27 of this SAP.	Matt Brookshire, MMEC Group
IIa & IIb	Sampling instrument decontamination records	Establish that proper decontamination procedures were implemented by field sampling personnel.	Matt Brookshire, MMEC Group
IIa	Sampling instrument calibration logs	Establish that field instrumentation requiring calibration was implemented in accordance with the method, manufacturer's manual, or procedure.	Matt Brookshire, MMEC Group
IIa	Chain of custody forms	Review COC records for completeness and accuracy on a daily basis. The QCM or assignee will look primarily for project information, sample analyses requested, number of field QC samples collected, and Level III and IV validation to be performed by the data validator.	Matt Brookshire, MMEC Group

SAP WORKSHEET #35 CONTINUED

Step IIa/IIb[1]	Validation Input	Description	Responsible for Validation
IIa	Sample receipts	Check the sample cooler for compliance with temperature and packaging requirements in Worksheet #19 of this SAP.	TestAmerica
IIa	Sample logins	Review sample login for accuracy against the COC form.	Matt Brookshire, MMEC Group TestAmerica
IIb	Sampling plan and procedures	Evaluate whether sampling plan was executed as specified (i.e., the number, location, and type of field samples that were collected and analyzed as specified in the SAP). Evaluate whether sampling procedures were followed with respect to equipment and proper sampling support (i.e., techniques, equipment, decontamination, volume, preservation, temperature, etc.).	Matt Brookshire, MMEC Group
IIb	Project quantitation limits	Determine that quantitation limits were achieved as outlined in the SAP and that the laboratory successfully analyzed a standard at the quantitation limit.	Linda Rauto, LDC TestAmerica
IIb	Performance criteria	Evaluate QC data against project-specific performance criteria in the SAP (i.e., evaluate quality parameters beyond those outlined in the methods).	Matt Brookshire, MMEC Group Linda Rauto, LDC
IIa	Laboratory data packages	The analytical laboratory will validate laboratory data packages for technical accuracy. Review data packages for accuracy against the laboratory data that was faxed/e-mailed.	TestAmerica
IIb	Data validation reports	Review data validation reports in conjunction with the project DQOs and DQIs per the SAP.	Matt Brookshire, MMEC Group

Notes:

1. IIa=compliance with methods, procedures, and contracts (see Table 10, page 117, UFP-QAPP manual, V.1, March 2005)
   IIb=comparison with measurement performance criteria in the SAP (see Table 11, page 118, UFP-QAPP manual, V.1, March 2005)

SAP WORKSHEET #36 ANALYTICAL DATA VALIDATION (STEPS IIA AND IIB) SUMMARY TABLE

Step IIa/IIb[1]	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa	Groundwater		In accordance with: • U.S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (2013) • U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (2010a) • DoD Quality Systems Manual Version 5.0 (2013) • NAVFAC SW EWI #1 Data Validation Guidelines for Chemical Analysis of Environmental Samples (2001) • U.S. EPA SW-846 Methods	Linda Rauto, LDC
IIb	Groundwater		In accordance with: • U.S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (2013) • U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (2010) • DoD Quality Systems Manual Version 5.0 (2013) • NAVFAC SW EWI #1 Data Validation Guidelines for Chemical Analysis of Environmental Samples (2001) • U.S. EPA SW-846 Methods	TestAmerica

Notes:

1. IIa=compliance with methods, procedures, and contracts (see Table 10, page 117, UFP-QAPP manual, V.1, March 2005)
   IIb=comparison with measurement performance criteria in the SAP (see Table 11, page 118, UFP-QAPP manual, V.1, March 2005)

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SAP WORKSHEET #37 USABILITY ASSESSMENT

The usability assessment process is used to evaluate and document the usability (i.e., Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity [PARCCS]) of the data by considering the project DQOs and whether the data are suitable during the decision-making process. The analytical laboratory will be responsible for reviewing all analytical data generated under this contract to ensure that they meet the requirements of this UFP-QAPP. Each analyst reviews the quality of his/her work based on established protocols specified in laboratory SOPs, analytical method protocol, project-specific requirements, and DQOs.

The intent of the data quality assessment process is to establish the levels of PARCCS and the usability of the final results with respect to the project DQOs. Upon completion of data validation, each data point will be assessed as nonqualified, qualified, or rejected based upon the acceptance criteria, and data validation flags will be added to the project database. These parameters will be based upon the analytical data quality and will encompass the QC criteria established in this UFP–QAPP. Qualification will be given according to each sample's sample delivery group, and will be based on the guidelines as presented in the National Functional Guidelines for Data Review (U.S. EPA, 2013; 2010a). Both analytical completeness and contract compliance completeness levels will then be determined for each analytical parameter. Finally, the overall usefulness of the data will be established as related to the project DQOs.

The usability assessment process will consist of reviewing the analytical data validation reports for both usable analytical data (i.e., no validation qualifications or estimated "J"/"UJ" qualifications) and rejected ("R" qualified) analytical data, as well as evaluating the field and analytical data for discrepancies or deviations. This assessment will evaluate the impact of the discrepancies or deviations on the usability of the data and assess whether all the necessary information has been provided for use in the decision-making process. The assessment will assess whether there were deviations in sampling activities (e.g., incorrect sample location or analysis performed), COC documentation, or holding times; compromised samples (i.e., damaged samples) and the need to resample; or changes to SOPs or methods that could potentially impact data quality. An evaluation of QC sample results will be performed to assess whether unacceptable QC results (e.g., blank contamination) impact data usability. Data use limitations will be discussed in the quality control reports for data that do not meet the DQOs or DQIs. Other parameters to be evaluated during the usability assessment may include, but are not limited to, the following:

- Matrix Effects – matrix conditions (e.g., salt water) that may impact the performance of the extraction or analytical method
- Site conditions – unusual weather conditions or site conditions that may affect the sampling plan
- Identification of critical and noncritical samples or target analytes
- Background or historical data
- Data restrictions – data that do not meet the project DQOs or were "R" qualified might be restricted but usable as qualitative values for limited decision-making purposes

SAP WORKSHEET #37 CONTINUED

Precision

Precision is the measure of variability between individual sample measurements under prescribed conditions. Analytical precision is the measurement of the variability associated with duplicate or replicate analyses. Laboratory duplicate, MSD, and LCS duplicate (if analyzed) samples will be used to assess field and analytical precision. The precision measurement will be determined using the relative percent difference (RPD) between the duplicate sample results as follows:

$$RPD = \frac{|A-B|}{(A+B)/2} \ x \ 100$$

where:

A = First duplicate concentration

B = Second duplicate concentration

The RPD limits for precision are presented in Worksheet #28. Associated samples that do not meet the criteria will be evaluated by the MMEC Group Project Chemist as described in Worksheet #35.

Accuracy

Accuracy is defined as the nearness of a result, or the mean of a set of results, to the true or accepted value. Analytical accuracy is measured by comparing the percent recovery (%R) of analytes spiked into a sample against a control limit. Accuracy will be measured using spiked samples, such as MS, MSD, LCS, and surrogates, if applicable. Surrogates, MS, MSD, and LCS analyzed for contaminants will also be used to assess matrix interferences. Calculation of %R is as follows:

$$Percent \ Recovery = \frac{S-C}{T} \ x \ 100$$

where:

S = Measured spike sample concentration

C = Sample concentration

T = True or actual concentration of the spike

The laboratory will review the QC samples and surrogate recoveries for each analysis to ensure that the %R lies within the control limits listed in the SAP. Otherwise, data will be flagged by the laboratory.

Representativeness

Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. It is a qualitative parameter that depends on proper design of the sampling program.

SAP WORKSHEET #37 CONTINUED

The representativeness of data will be maintained by the use of established field and laboratory procedures and their consistent application.

Field personnel will be responsible for collecting and handling samples according to the procedures in this SAP so that samples are representative of field conditions. Errors in sample collection, packaging, preservation, or COC procedures may result in samples being judged as non-representative and may form a basis for rejecting the data.

Completeness

Completeness is the percentage of measurements made that are judged to be valid compared to the amount that was expected to be obtained under correct, normal conditions. The completeness goal is to generate a sufficient amount of valid data to meet project needs. To be considered complete, the data set must contain all analytical results and data specified for the project. In addition, all data are compared to project requirements to determine whether specifications were met. Completeness is evaluated by comparing the project objectives to the quality and quantity of the data collected to determine if any deficiencies exist. Data validation and data quality assessment will determine which data are valid and which data are rejected or missing.

Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with a rejected (R) flag. The requirement of completeness is 90 percent for samples and is determined using the following equation:

$$Completeness = \frac{Number\ of\ Valid\ Measurements}{Total\ Number\ of\ Measurements}\ x\ 100$$

Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another, whether it was generated by a single laboratory or during inter-laboratory studies. The use of standardized field and analytical procedures ensures comparability of analytical data. Sample collection and handling procedures will adhere to U.S. EPA-approved protocols. Laboratory procedures will follow standard analytical protocols, use standard units and standardized report formats, follow the calculations as referenced in approved analytical methods, and use a standard statistical approach for QC measurements.

Sensitivity

Sensitivity is the ability of the analytical test method and/or instrumentation to differentiate between detector responses to varying concentrations of the target constituent. Methodology to establish sensitivity for a given analytical method or instrument includes examination of standardized blanks, instrument detection limit studies, and calibration of the quantitation limit (QL). The findings of the usability of the data relative to sensitivity will be included in the report, including any limitations on the data set and/or individual analytical results.

SAP WORKSHEET #37 CONTINUED

The PARCCS measured performance criteria are described in Worksheets #12, 15, and 28. The following steps will be performed:

- Evaluate whether the project-required quantitation limits listed in Worksheet #15 were achieved for non-detected site contaminants. If no detectable results were reported and data are acceptable for the verification and validation steps, then the data are usable.
- If detectable concentrations are reported and the verification and validation steps are acceptable, the data are usable.
- If verification and validation are not acceptable, the data are qualified. Qualifiers (J, UJ) will be added for minor QC deviations that do not affect the data usability, or rejection (R) will be added for major QC deviations affecting data usability. The impact of rejected data will be evaluated and resampling may be necessary. The use of estimated data will be discussed in the project report.
- For statistical comparisons and mathematical manipulations, non-detect values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate will be used to represent the concentration at that sample location.
- Statistical tests will be conducted to identify potential outliers. Potential outliers will be removed if a review of the field and laboratory documentation indicates that the results are true outliers.

Evaluative Procedures Used to Assess Overall Measurement Error Associated With the Project

After completion of the data validation and review of the data quality indicator, the data will be reconciled with the measured performance criteria to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and statistical analyses will be performed to estimate the set characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and proportion of samples with raised results. The data will be presented in a tabular format. These inspections and statistical analyses will be designed to:

- Identify deviations from the field sampling SOPs, from laboratory analytical methods, from this SAP, and from the validation process.
- Evaluate effects of the above-listed deviations from planned procedures on the quality of the data to meet project objectives.
- Identify elevated sample quantitation limits and explain the impact of these results on project objectives.
- Identify unusable, rejected data.
- Evaluate project assumptions.

SAP WORKSHEET #37 CONTINUED

- Identify unanticipated data set characteristics such as laboratory variance greater than the sampling variance (i.e., ANOVA, t-test), if enough data are available.
- Identify and evaluate potential data outliers (95 percent confidence goodness-of-fit test on probability plot data); the plotted data will be transformed, if necessary, depending on the observed distribution.
- Evaluate adherence to investigation objectives and decision rules.
- Ensure completion of any corrective actions (CAs).
- Identify any data gaps.

Personnel Responsible for Performing the Usability Assessment

MMEC Group Project Chemist or designee.

Documentation to be Generated During Usability Assessment (and how usability assessment results will be presented so that they identify trends, relationships [correlations], and anomalies)

The data will be presented in tabular format; data qualifiers such as estimation (J, UJ) or rejection (R) will be applied. Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest resampling if necessary to fill out the data gaps.

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Figures

Figure A-1	Facility Location Map
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Figure A-3	Groundwater Monitoring Well Locations
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Figure A-6	OU – 1 IR SITE 7
Figure A-7	OU – 1 IR SITE 8
Figure A-8	OU – 1 IR SITE 14
Figure A-9	OU – 1 IR SITE 16
Figure A-10	OU – 2A IR SITE 13
Figure A-11	OU – 2B IR SITE 3
Figure A-12	OU – 2B IR SITE 4
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Figure A-15	OU – 2C IR SITES 5, 10, AND 12
Figure A-16	OU – 3 IR SITES 1 AND 32
Figure A-17	OU – 4A IR SITE 2
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Figure A-24	IR SITE 34
Figure A-25	IR SITE 35
Figure A-26	Soil Gas Monitoring Locations
Figure A-27	Project Schedule

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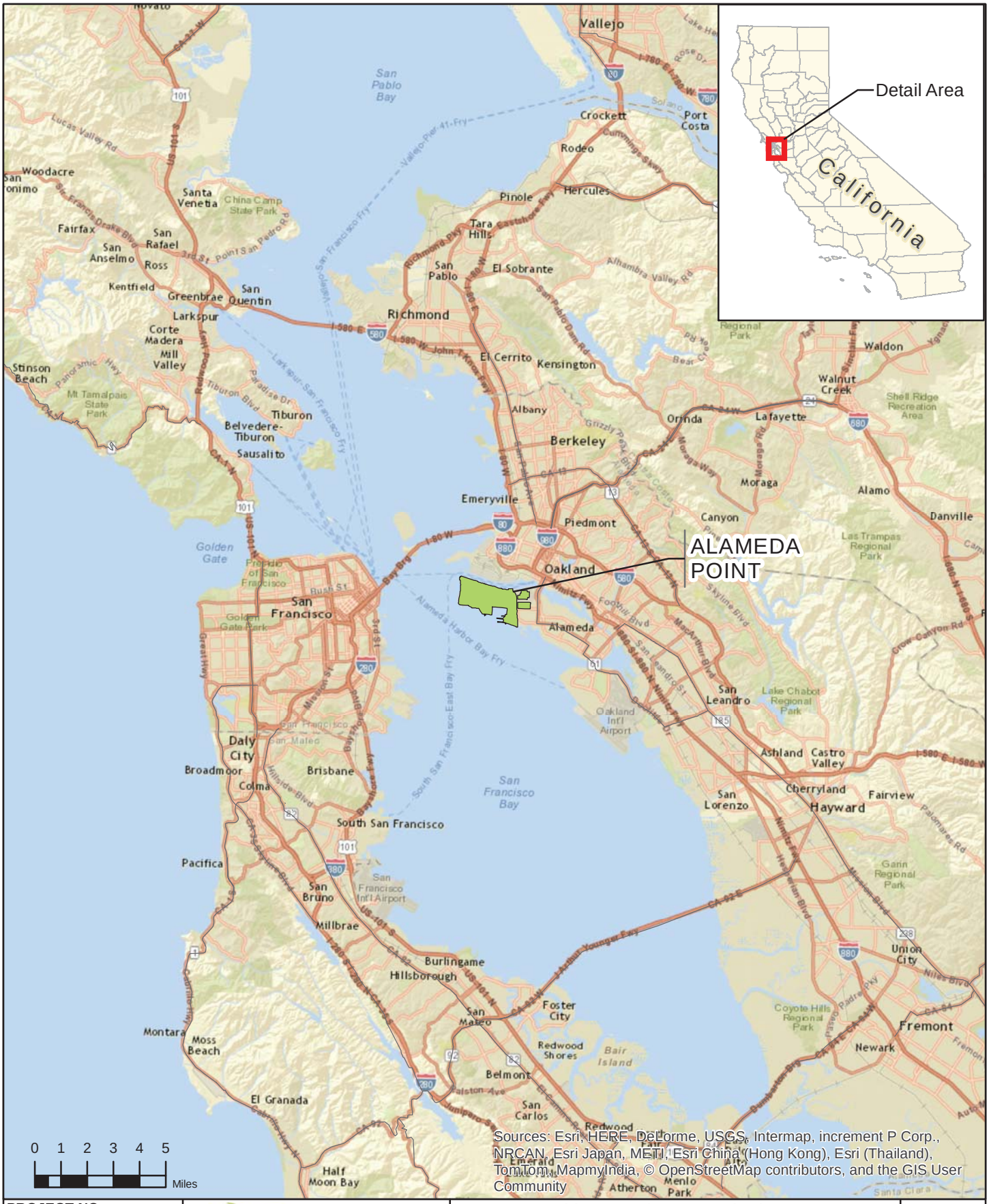
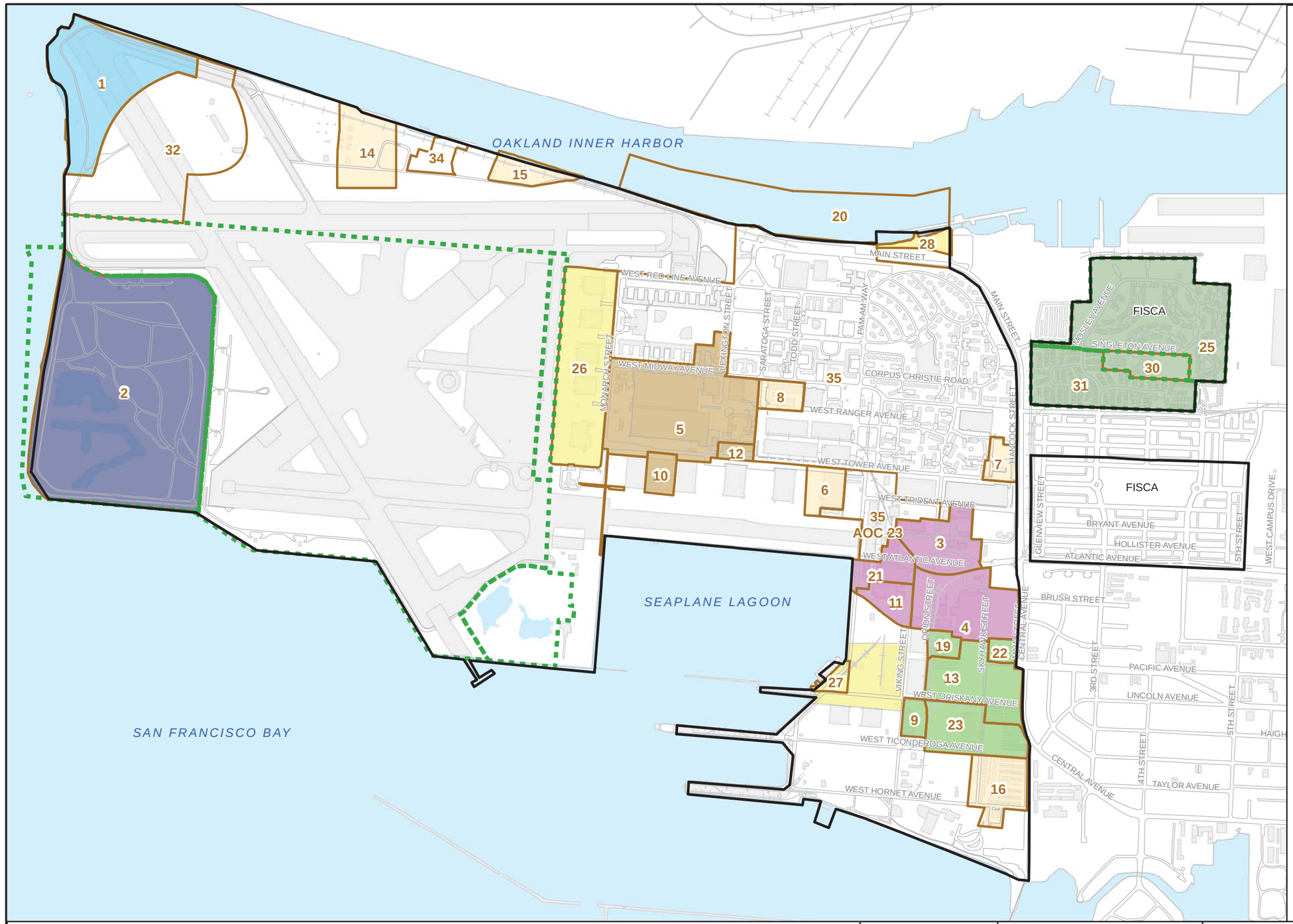


Sources: Esri, HERE, DeLorme, USGS, Intermap, increment P Corp., NRCAN, Esri Japan, METI, Esri China (Hong Kong), Esri (Thailand), TomTom, MapmyIndia, © OpenStreetMap contributors, and the GIS User Community

PROJECT NO.: 5023-14-6096	MEC GROUP	Basewide Groundwater Monitoring Program Alameda Point, Alameda, California	FIGURE
DATE: FEBRUARY 2015		FACILITY LOCATION MAP	A-1
DRAWN BY: SB			
CHECKED BY: KO			



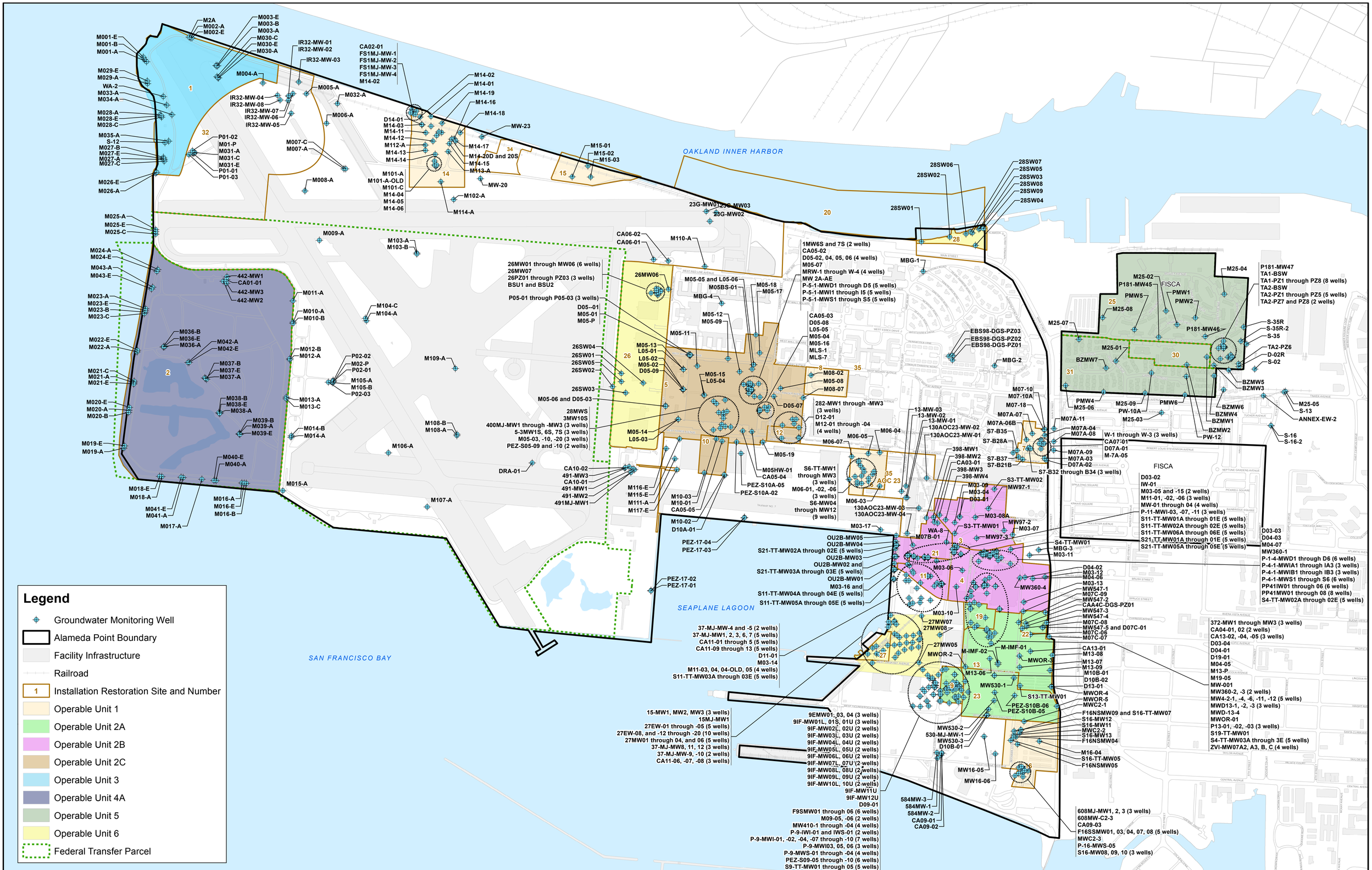
Legend

- Alameda Point Boundary
- Facility Infrastructure
- Railroad
- 1 Installation Restoration Site and Number
- Operable Unit 1
- Operable Unit 2A
- Operable Unit 2B
- Operable Unit 2C
- Operable Unit 3
- Operable Unit 4A
- Operable Unit 5
- Operable Unit 6
- Federal Transfer Parcel

Notes:

FISCA = Fleet and Industrial Supply Center Alameda

PROJECT NO.: 5023-14-6096	MMEC GROUP	Basewide Groundwater Monitoring Program Alameda Point, Alameda, California	FIGURE
DATE: FEBRUARY 2015		OPERABLE UNIT AND INSTALLATION RESTORATION SITE LOCATIONS	A-2
DRAWN BY: SB			
CHECKED BY: KO			



Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH (6 wells were not plotted on the map because coordinates were unavailable).
Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014.
Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.
FISC = Fleet and Industrial Supply Center Alameda

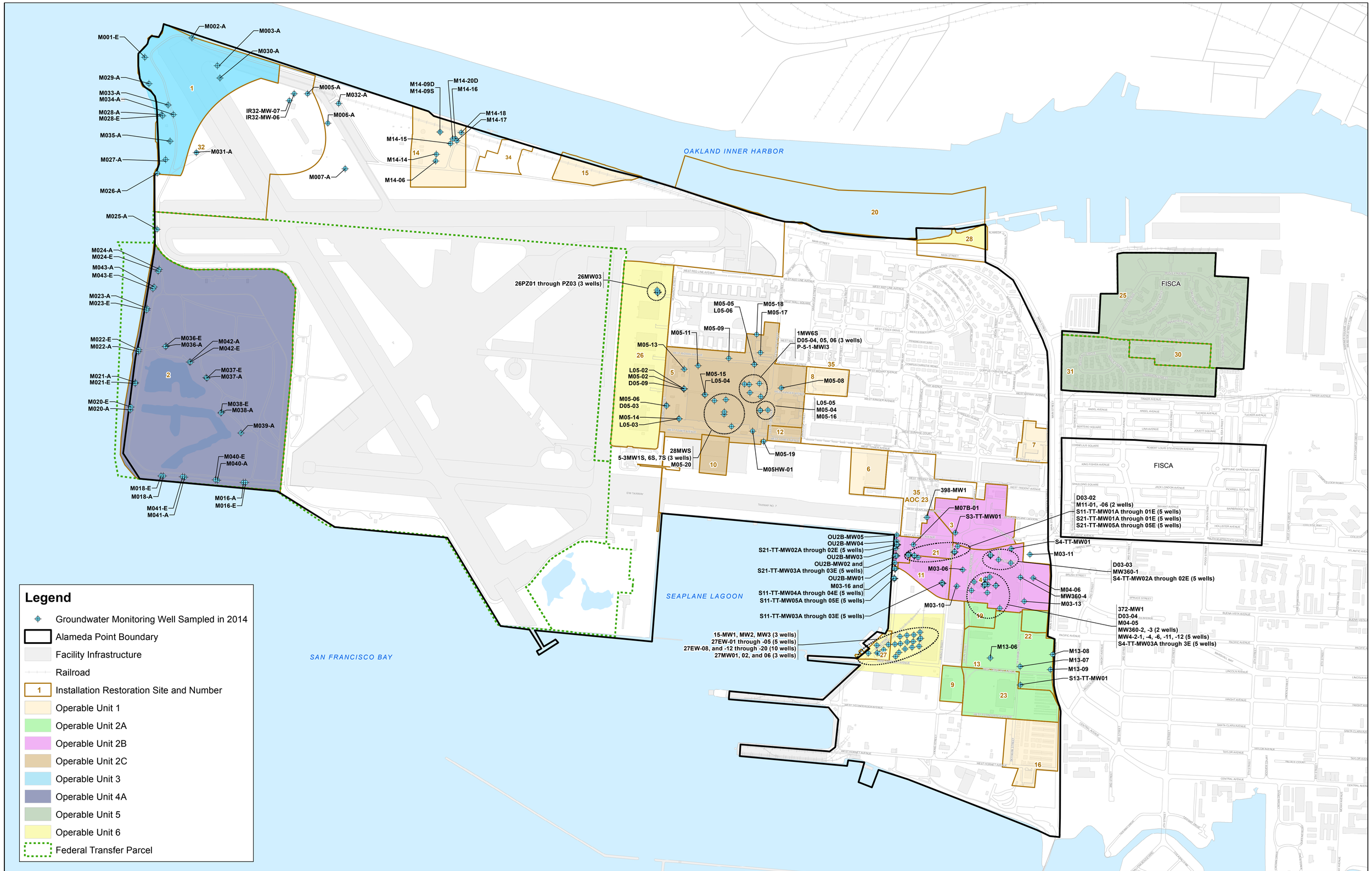
PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

GROUNDWATER MONITORING WELL LOCATIONS

FIGURE A-3



Notes:
Only the wells sampled during the 2014 Basewide Groundwater Sampling Program are shown. Refer to Figure A-3 for the locations of all wells.
Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014.
FISC = Fleet and Industrial Supply Center Alameda

PROJECT NO.:	0023-14-6096
DATE:	FEBRUARY 2015
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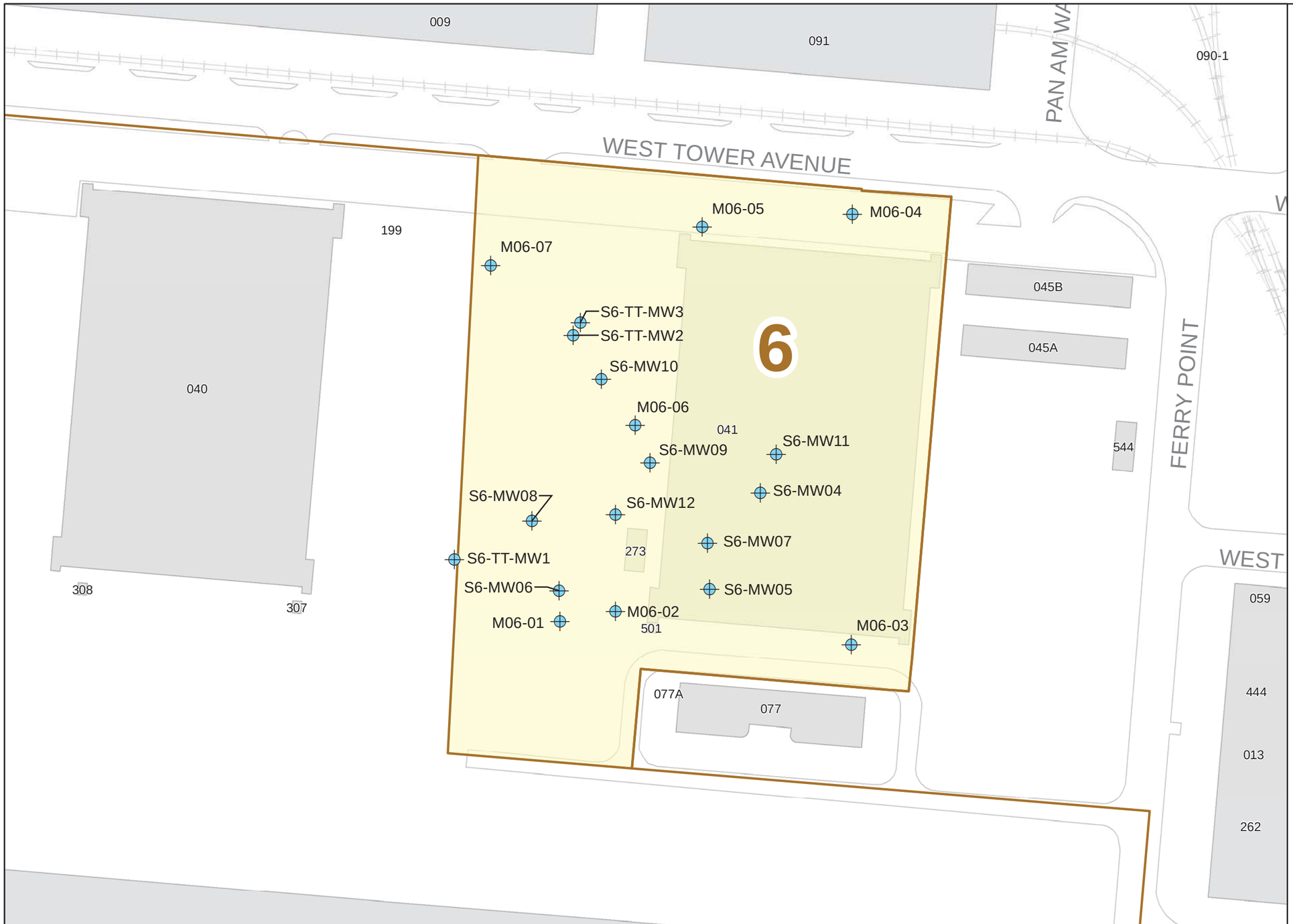
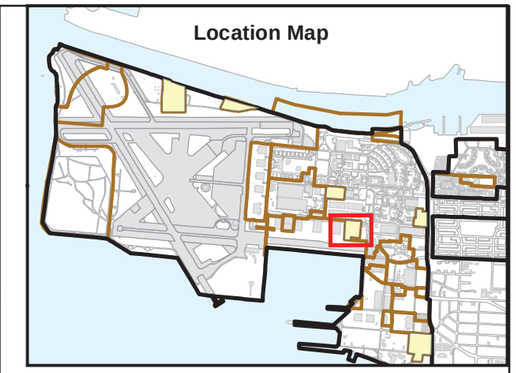
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

GROUNDWATER MONITORING WELLS SAMPLED IN 2014

FIGURE A-4





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 6 Installation Restoration Site and Number
- Operable Unit 1
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

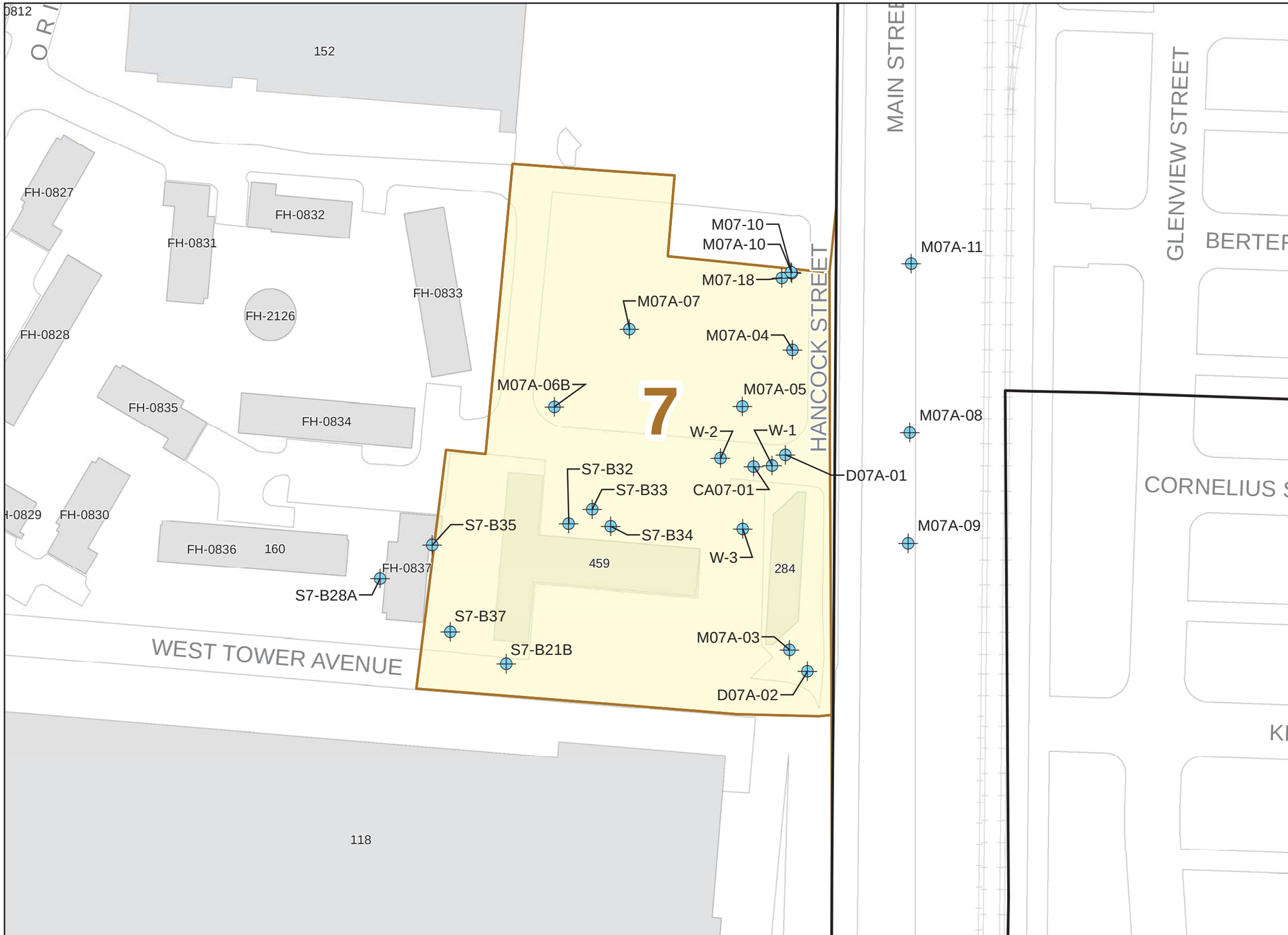
PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

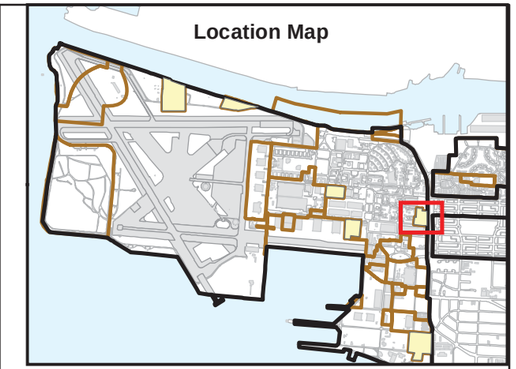
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 1
IR SITE 6

FIGURE
A-5





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 7 Installation Restoration Site and Number
- Operable Unit 1
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

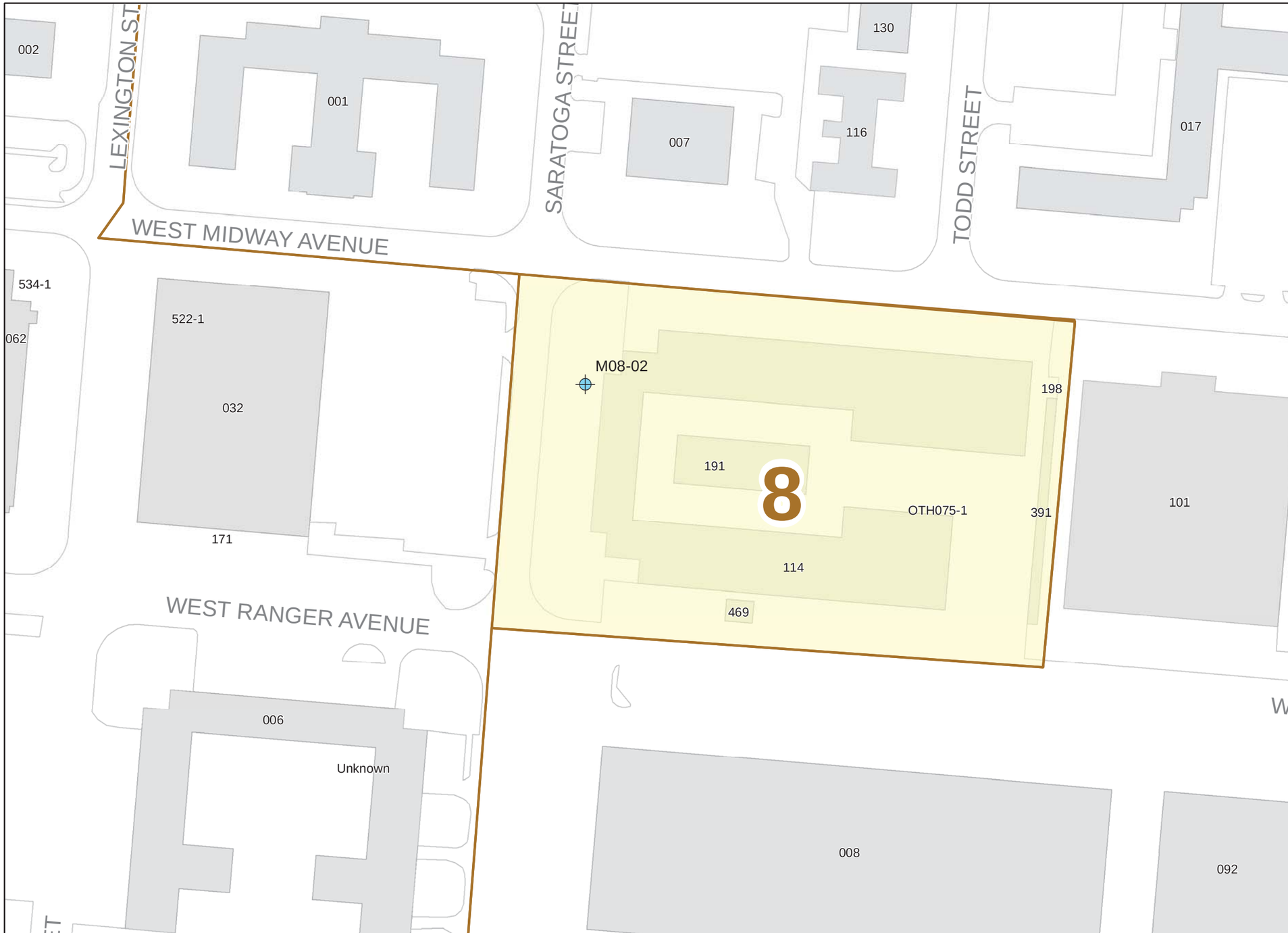
MNEC GROUP

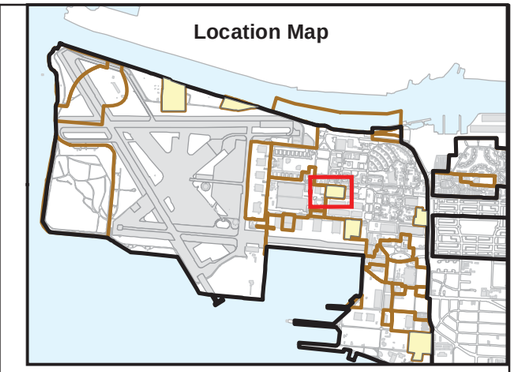
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 1
IR SITE 7

FIGURE

A-6





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 8 Installation Restoration Site and Number
- Operable Unit 1
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

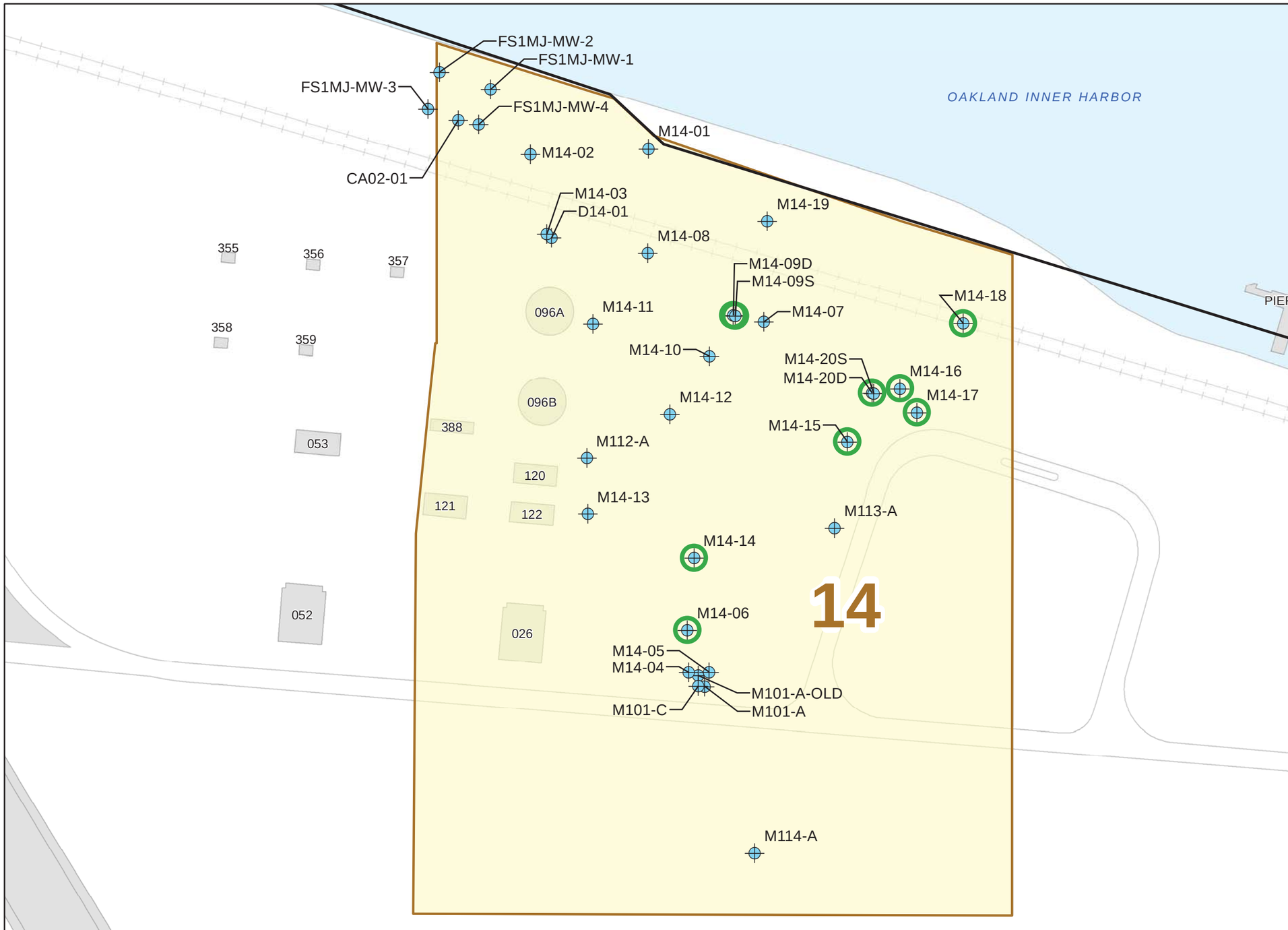
PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

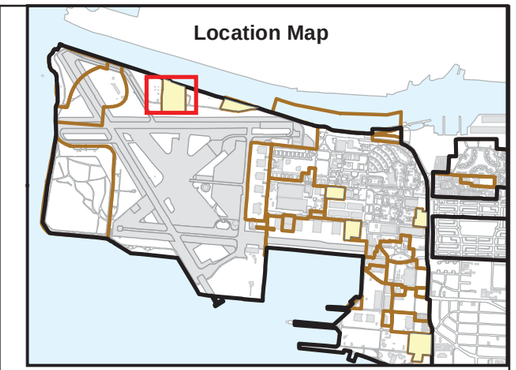
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 1
IR SITE 8

FIGURE
A-7





Legend

- Groundwater Monitoring Well
- Wells Sampled in 2014
- Alameda Point Boundary
- 14 Installation Restoration Site and Number
- Operable Unit 1
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 120 240 Feet

N

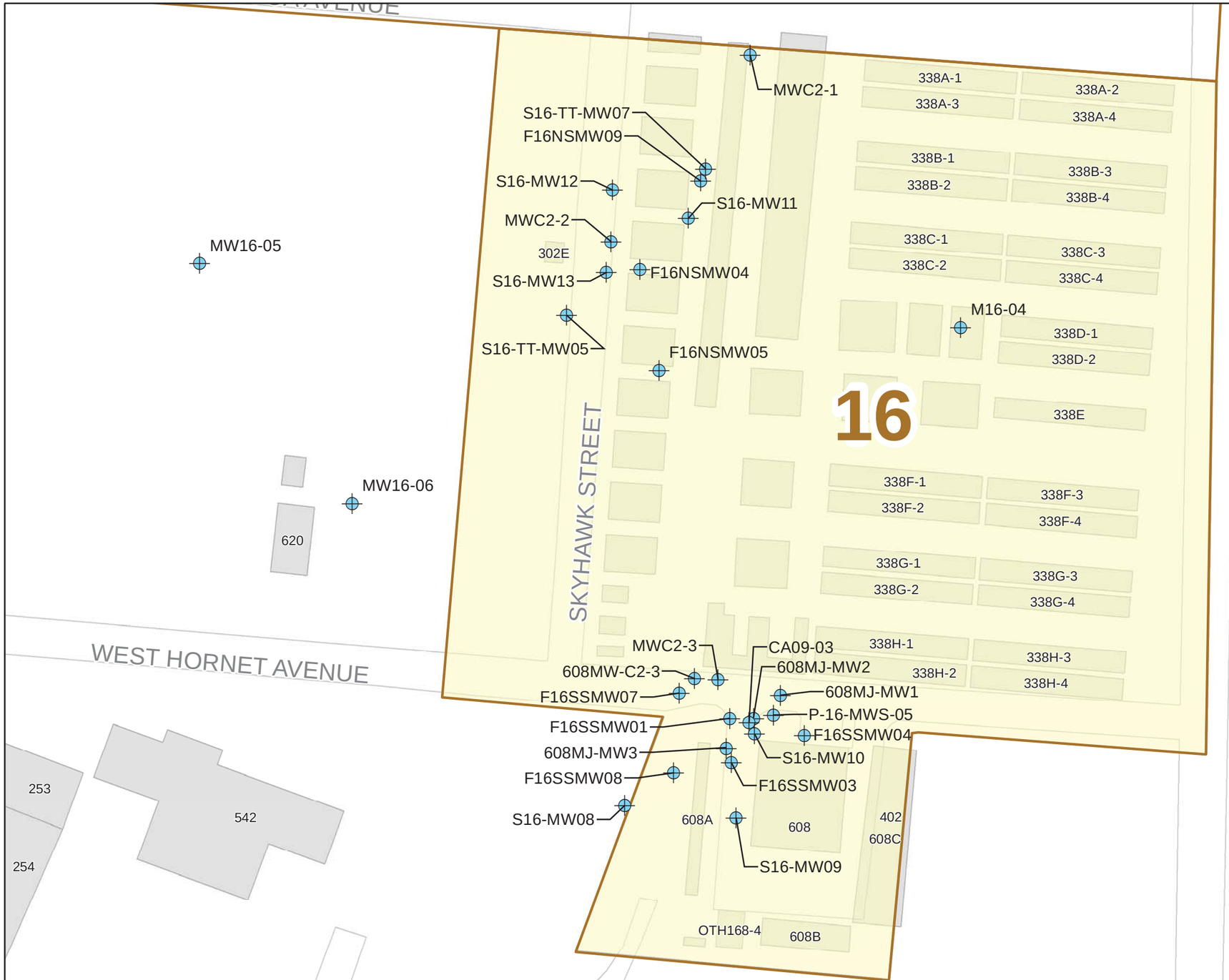
PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

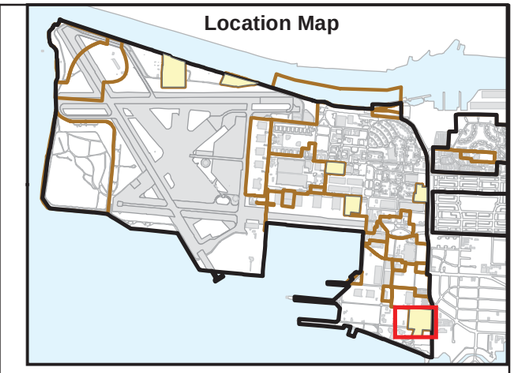
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 1
IR SITE 14

FIGURE
A-8





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 16 Installation Restoration Site and Number
- Operable Unit 1
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014. Well P-16-IWS-03 is not shown because the survey coordinates are unknown.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

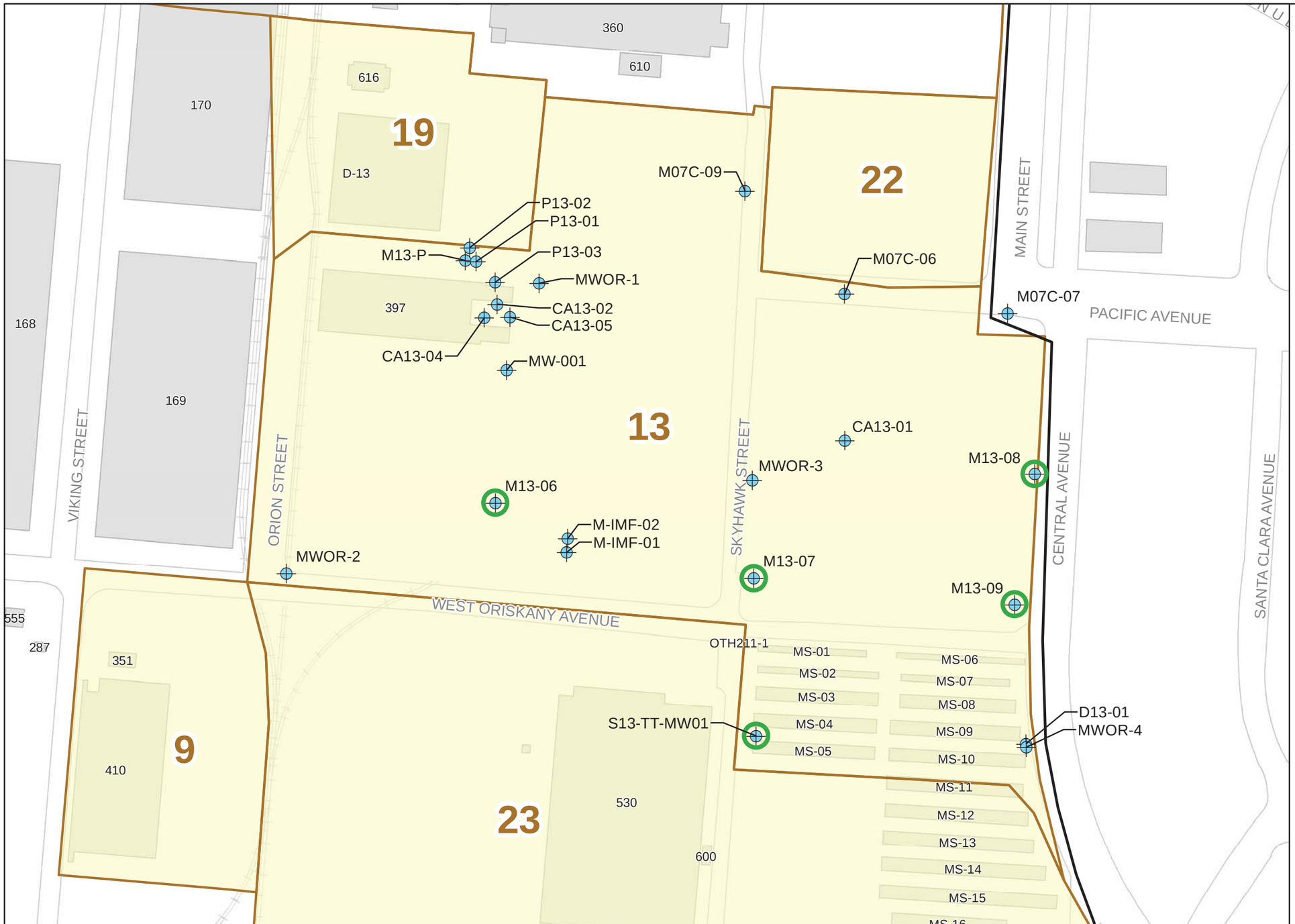
MMEC GROUP

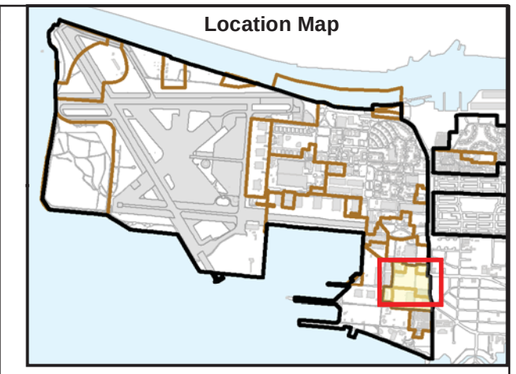
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 1
IR SITE 16

FIGURE

A-9





Legend

- Groundwater Monitoring Well
- Wells Sampled in 2014
- Alameda Point Boundary
- 13 Installation Restoration Site and Number
- Operable Unit 2A
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 150 300 Feet

N

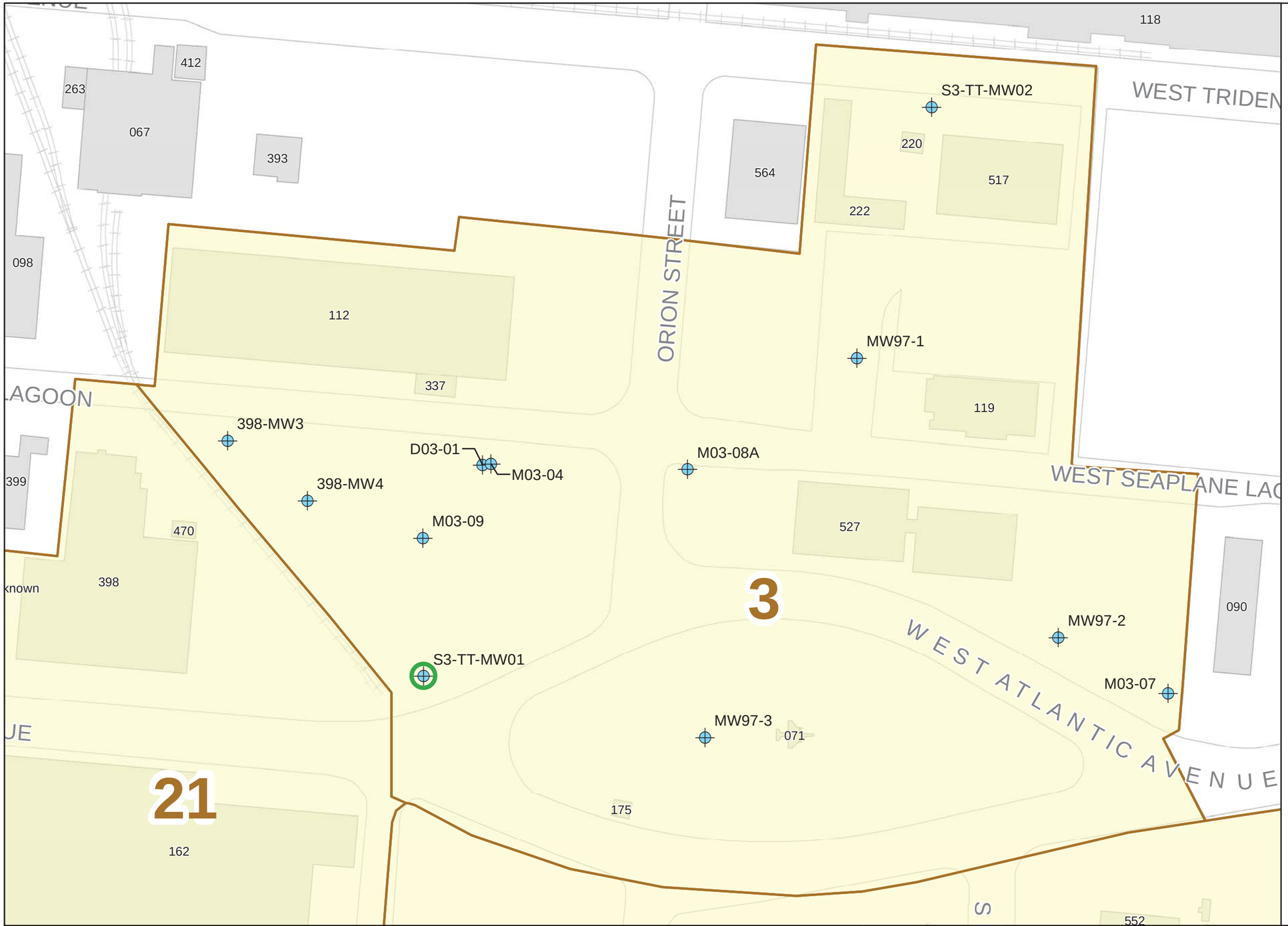
PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

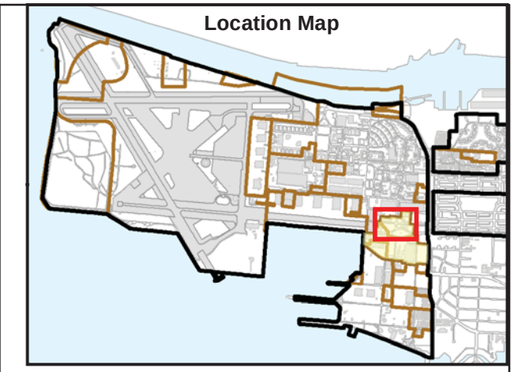
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 2A
IR SITE 13

FIGURE
A-10





Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 3 Installation Restoration Site and Number
- Operable Unit 2B
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

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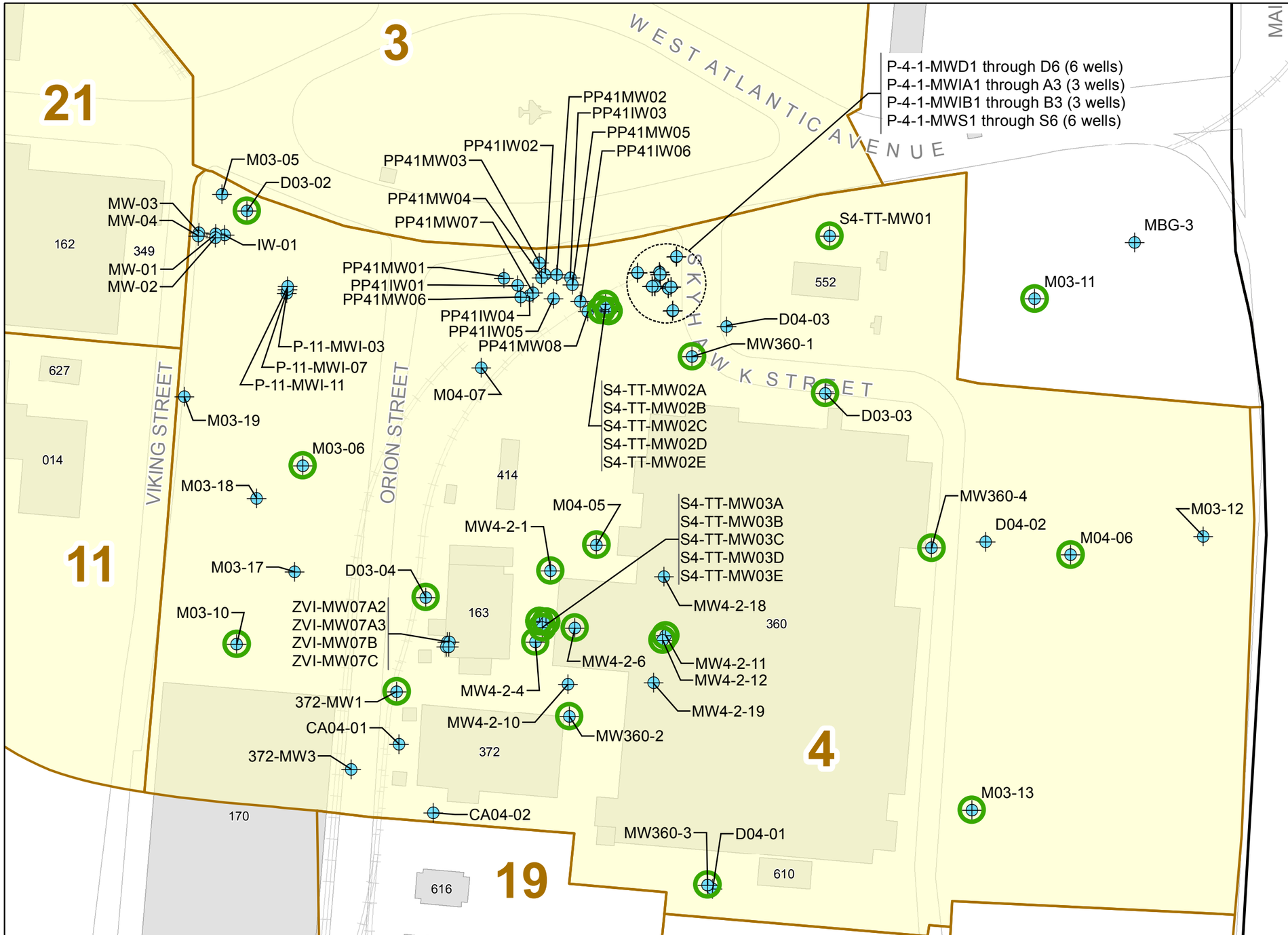
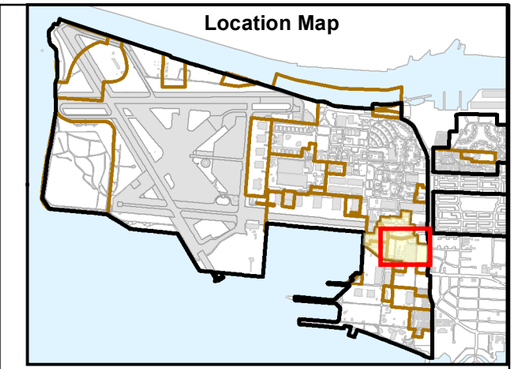
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 2B
IR SITE 3

FIGURE
A-11





Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 4 Installation Restoration Site and Number
- Operable Unit 2B
- Facility Infrastructure

Notes:
Well M011-01 is not shown because the survey coordinates are unknown.

All known and possibly existing groundwater monitoring wells and piezometers are shown (except as noted above), based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the basewide Groundwater Monitoring Program.

0 120 240 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	AUGUST 2015
DRAWN BY:	SB
CHECKED BY:	KO

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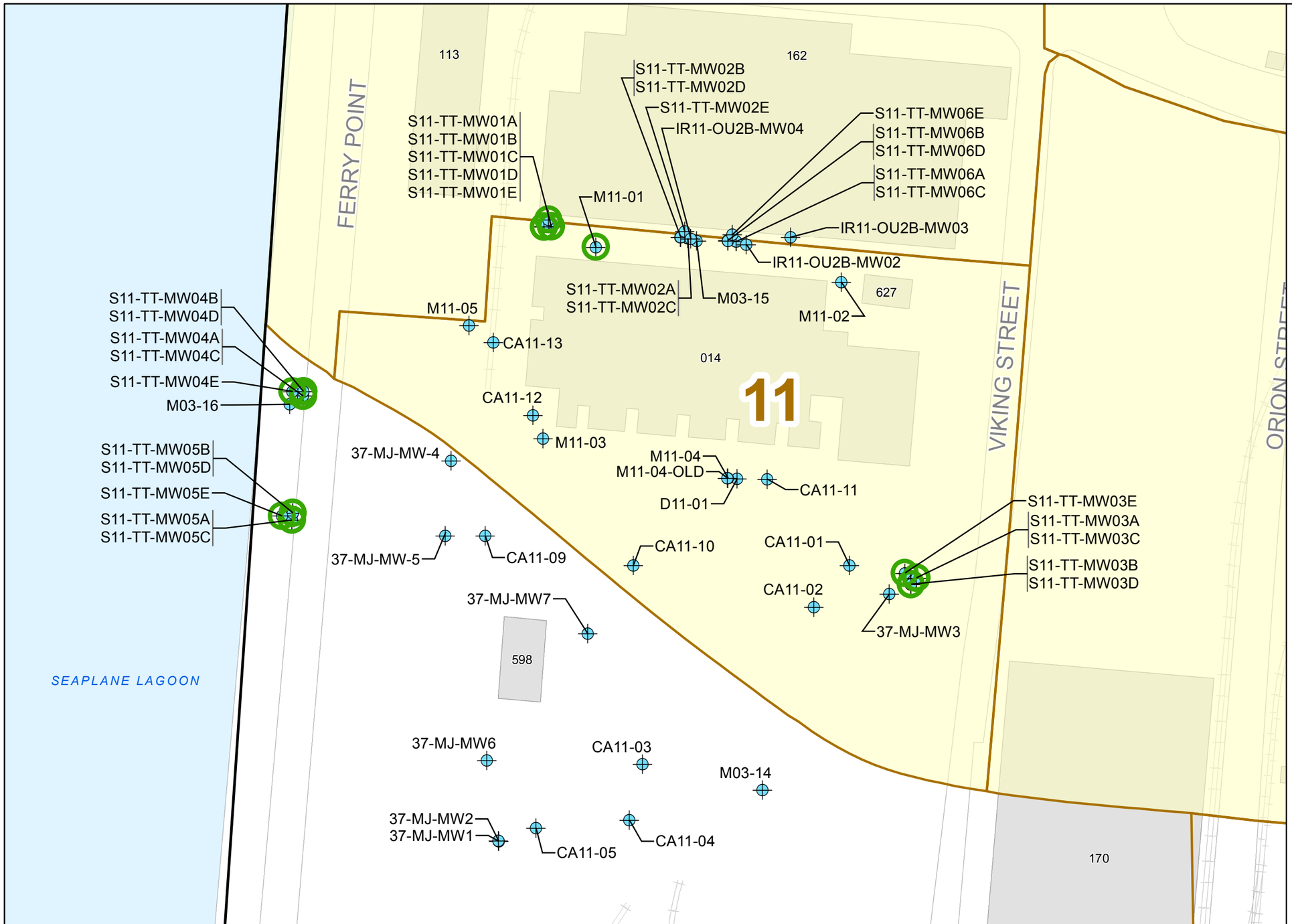
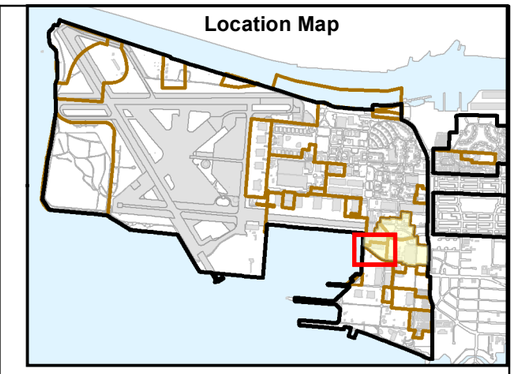
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 2B
IR SITE 4

FIGURE
A-12





Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 11 Installation Restoration Site and Number
- Operable Unit 2B
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

PROJECT NO.:	5023-14-6096
DATE:	AUGUST 2015
DRAWN BY:	SB
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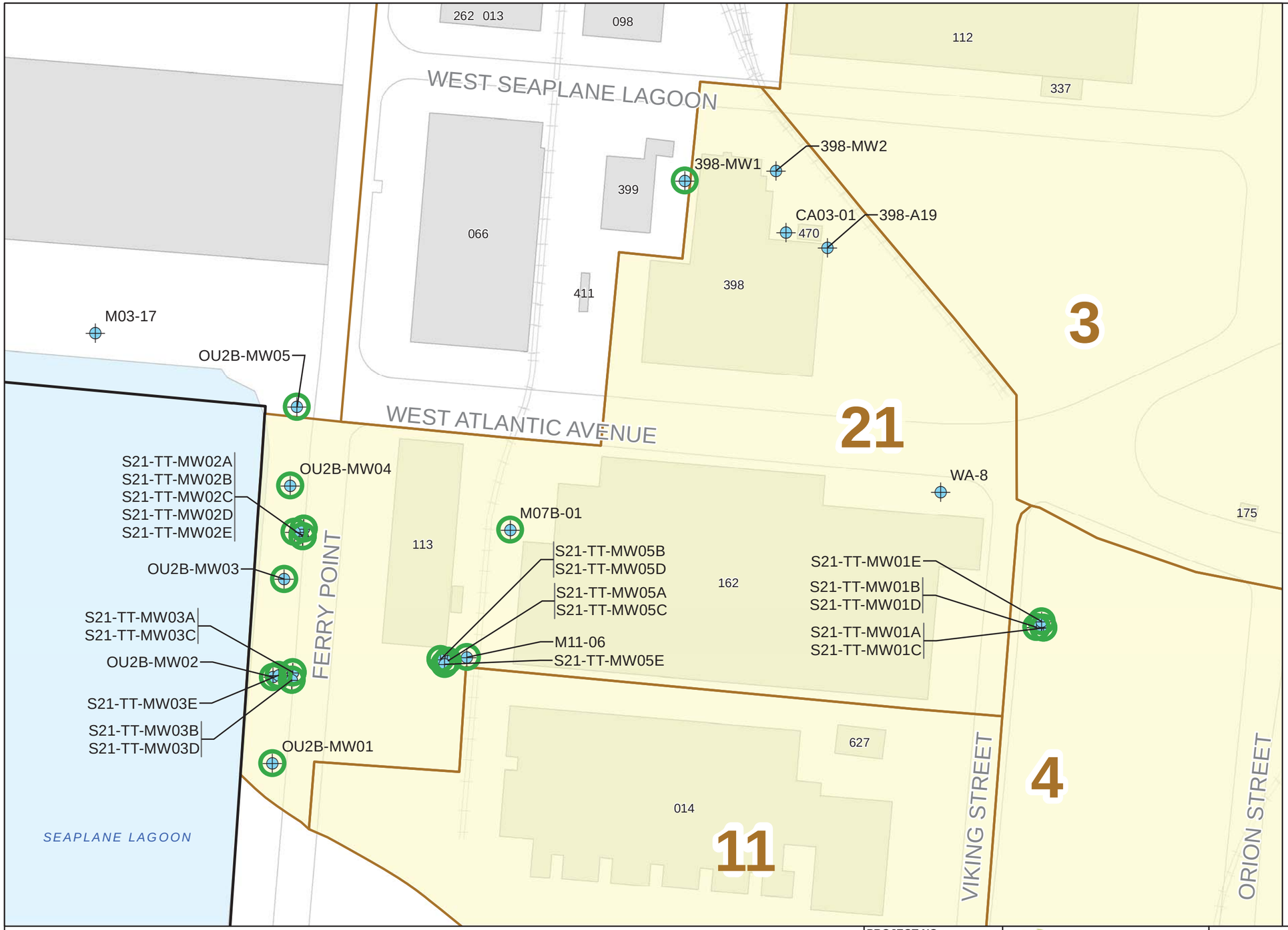
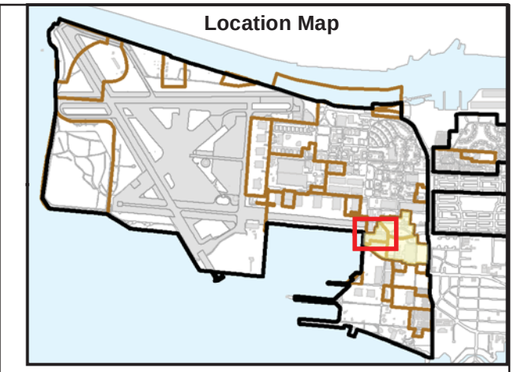
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 2B
IR SITE 11

FIGURE
A-13





Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 21 Installation Restoration Site and Number
- Operable Unit 2B
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

PROJECT NO.:	5023-14-6096
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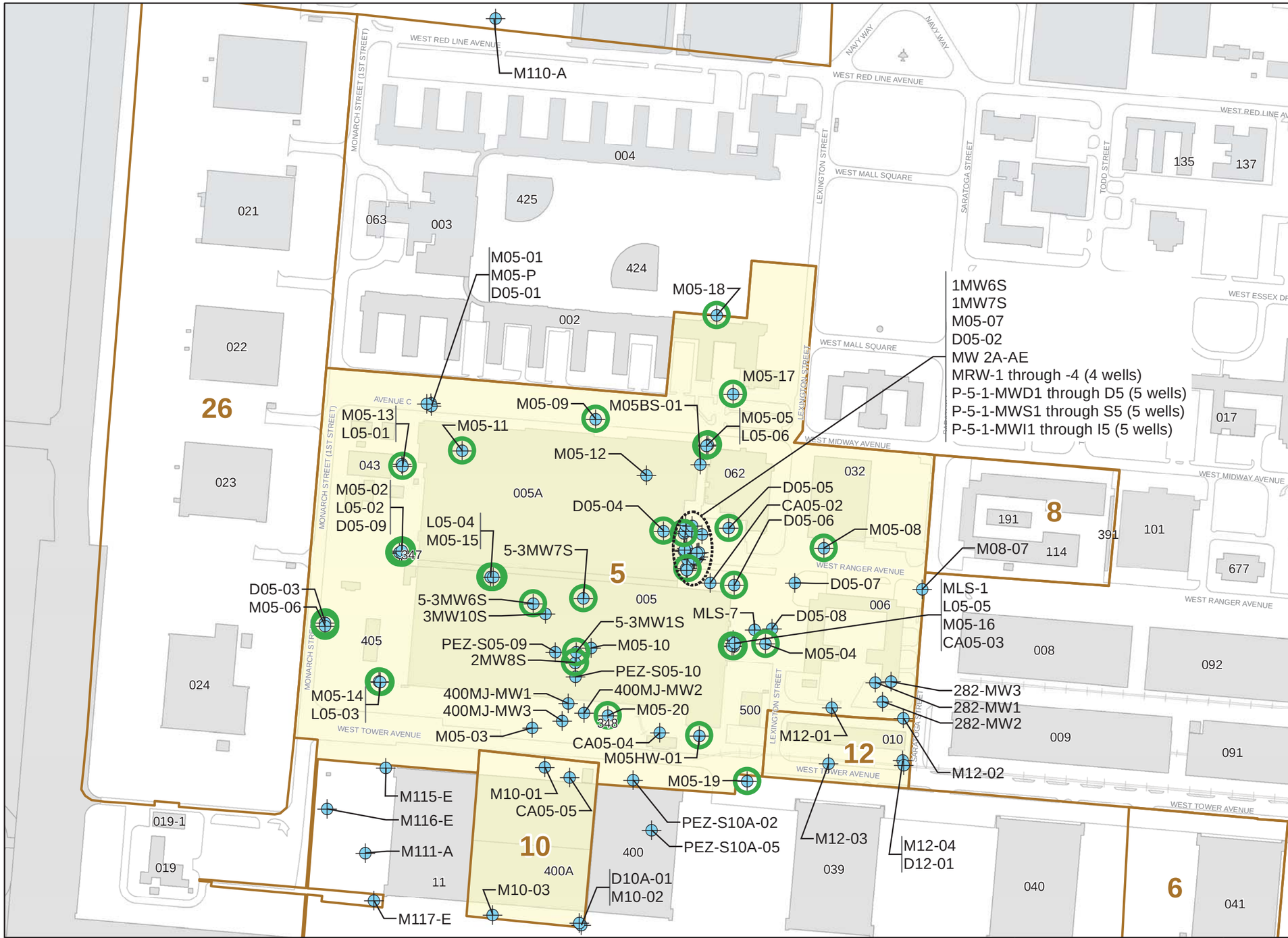
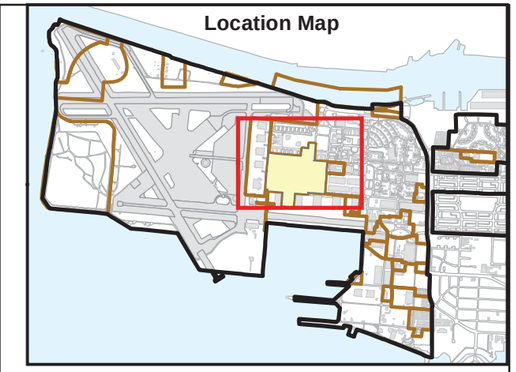
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 2B
IR SITE 21

FIGURE
A-14





Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 5 Installation Restoration Site and Number
- Operable Unit 2C
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 300 600 Feet

N

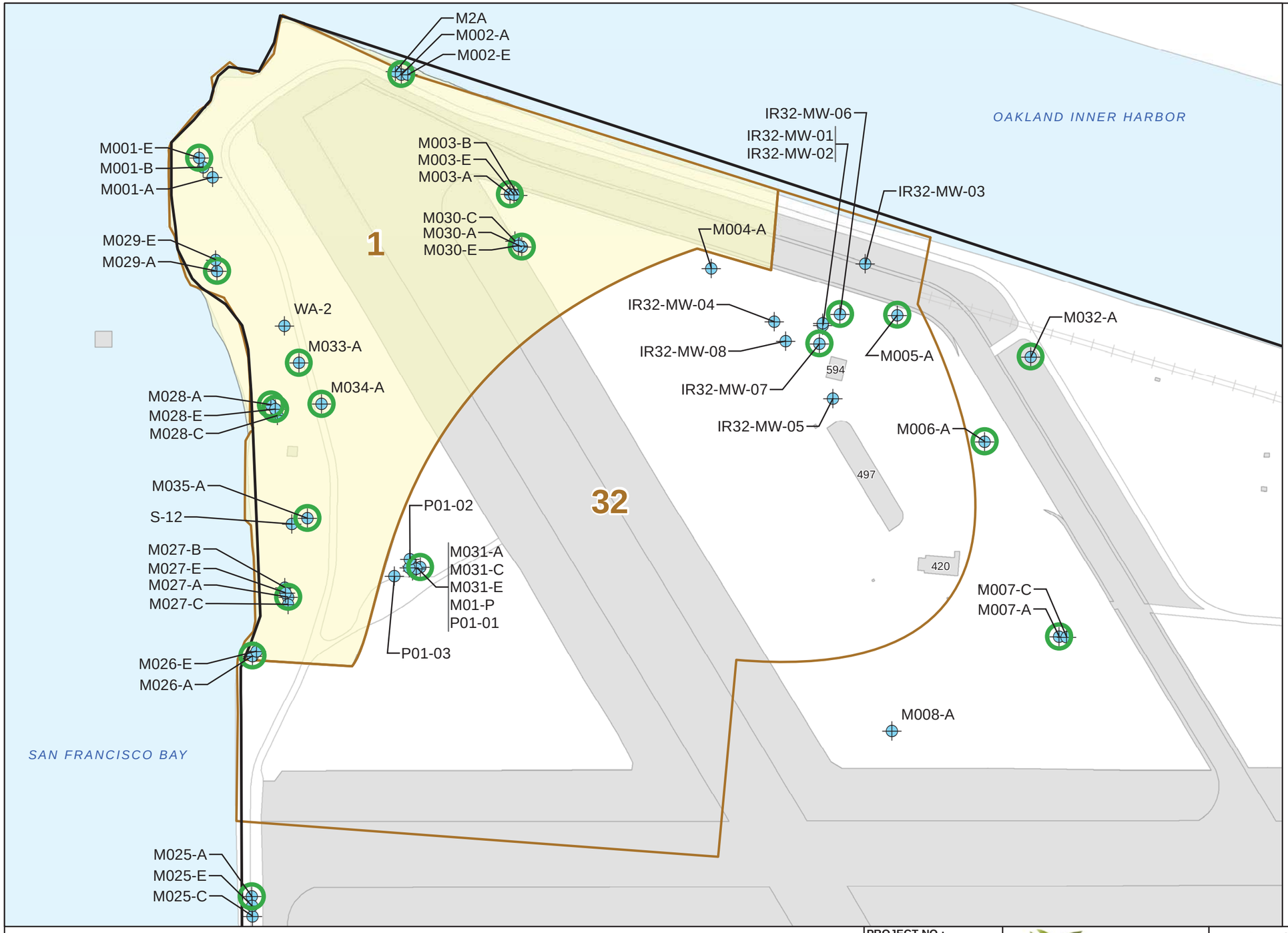
PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
CHECKED BY:	KO

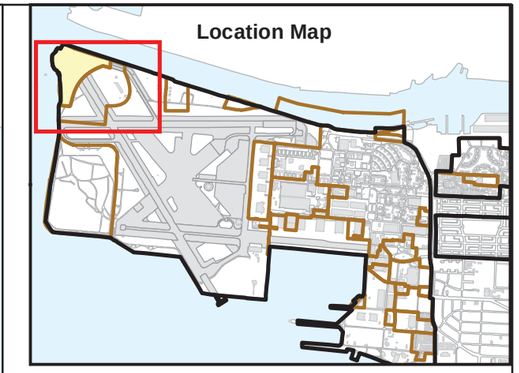
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 2C
IR SITES 5, 10, AND 12

FIGURE
A-15





Legend

- Groundwater Monitoring Well
- Wells Sampled in 2014
- Alameda Point Boundary
- 1 Installation Restoration Site and Number
- Operable Unit 3
- Facility Infrastructure

Notes:
A remedial action is on-going at IR Site 1 and the current monitoring well network shown will no longer be applicable after remedial action completion. An updated well network is anticipated in late Spring 2015.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 300 600 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
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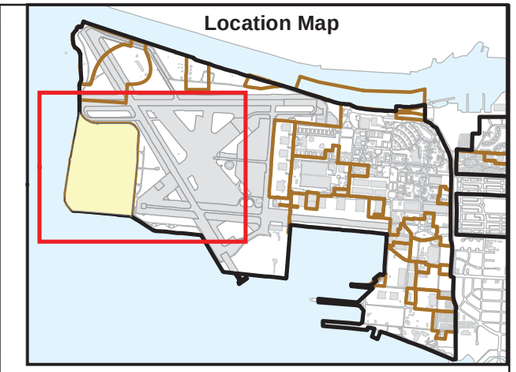
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 3
IR SITES 1 AND 32

FIGURE
A-16



Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 2 Installation Restoration Site and Number
- Operable Unit 4A
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 500 1,000 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
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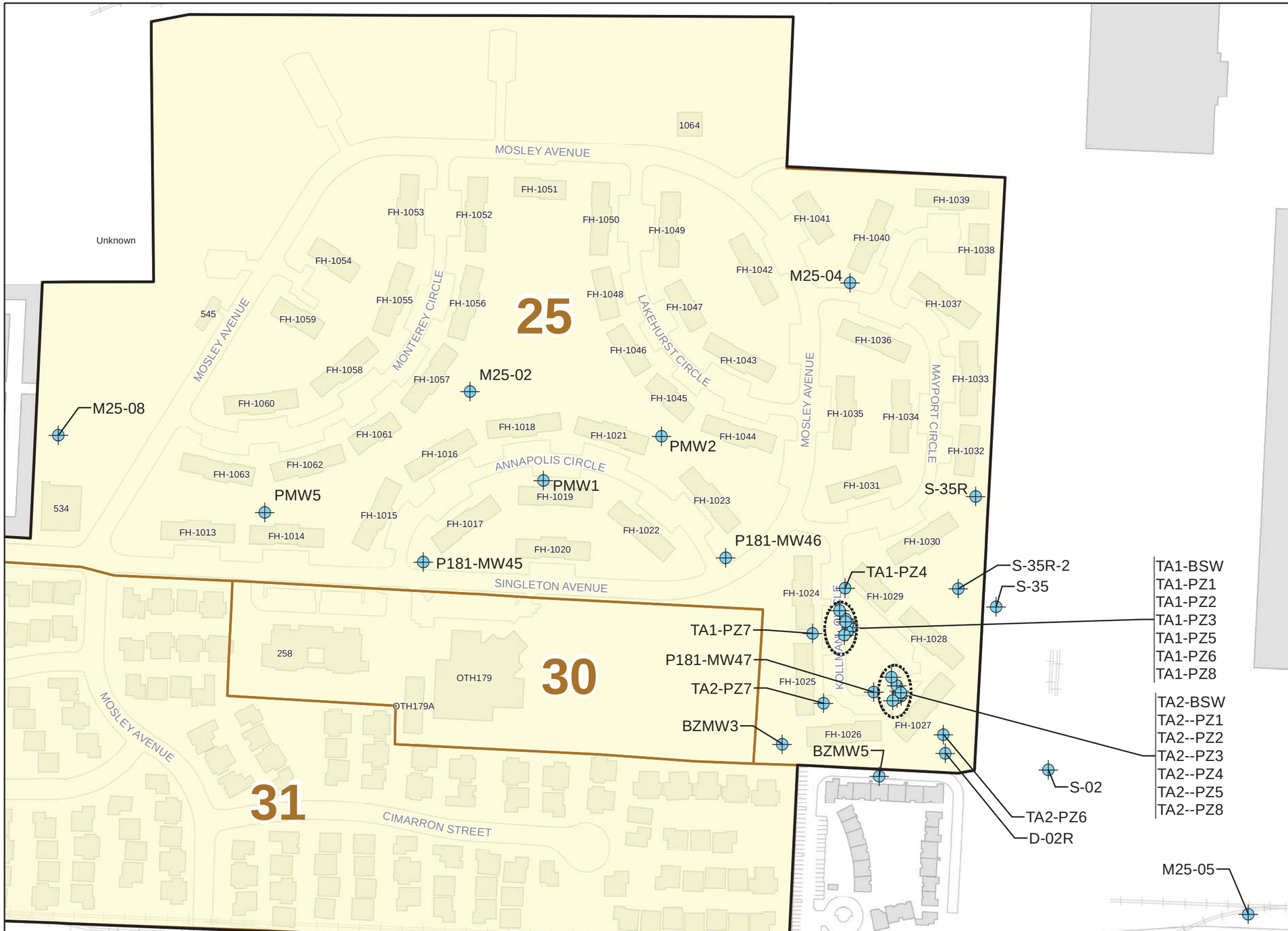
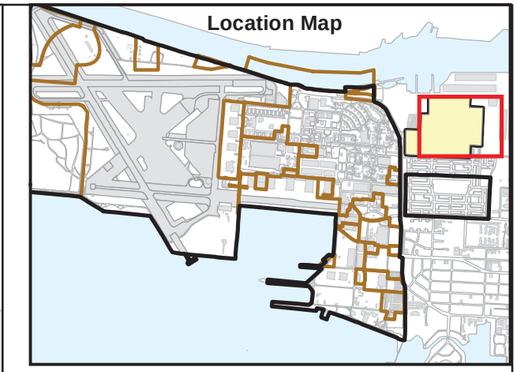
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 4A
IR SITE 2

FIGURE
A-17





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 25 Installation Restoration Site and Number
- Operable Unit 5
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 200 400 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
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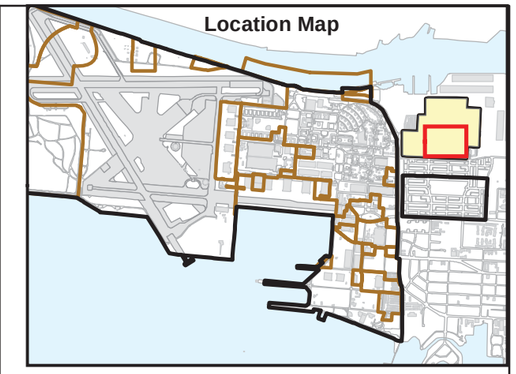
CHECKED BY:	KO

MNEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 5
IR SITE 25

FIGURE
A-18



Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 30 Installation Restoration Site and Number
- Operable Unit 5
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

PROJECT NO.:	5023-14-6096
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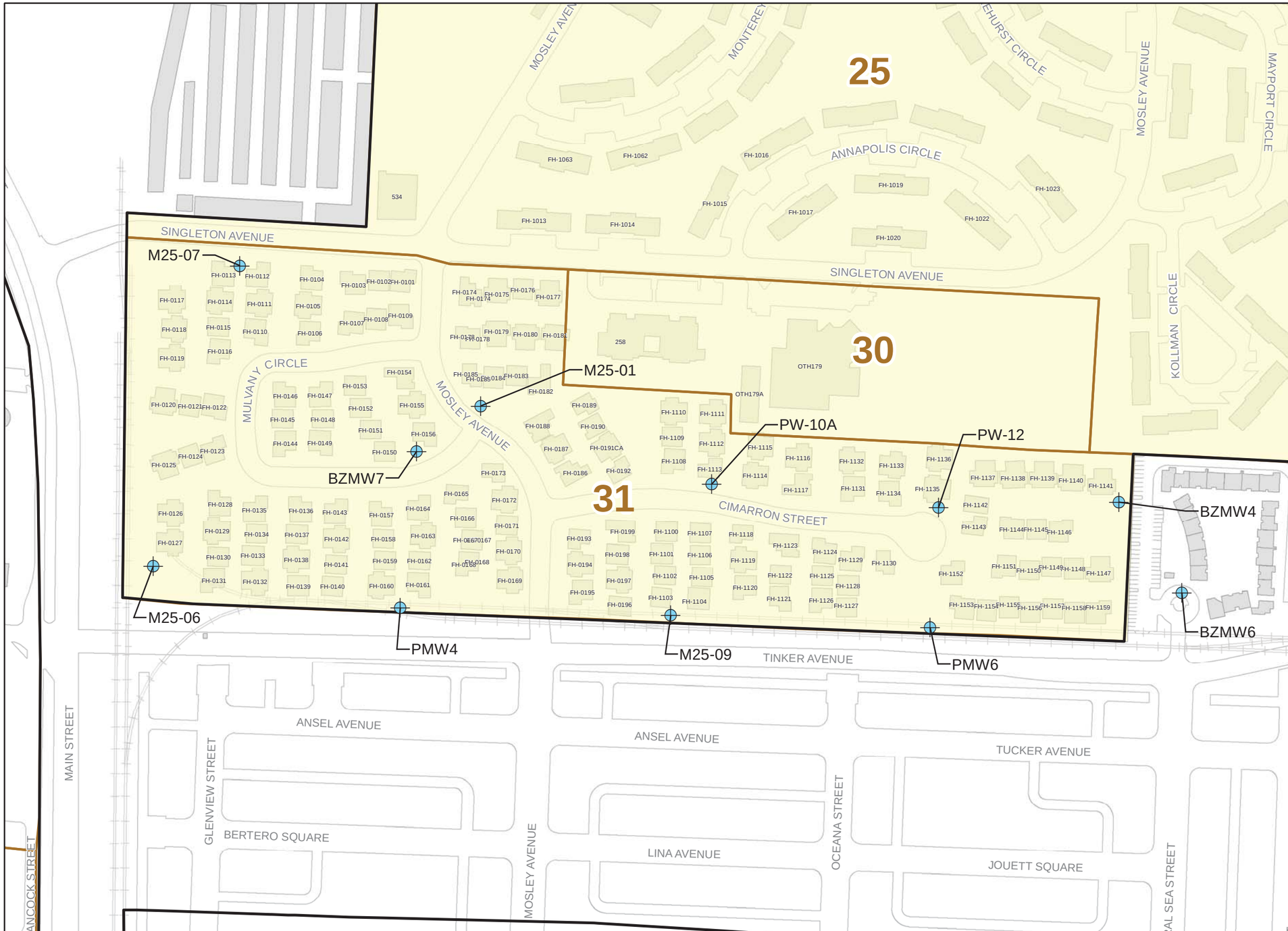
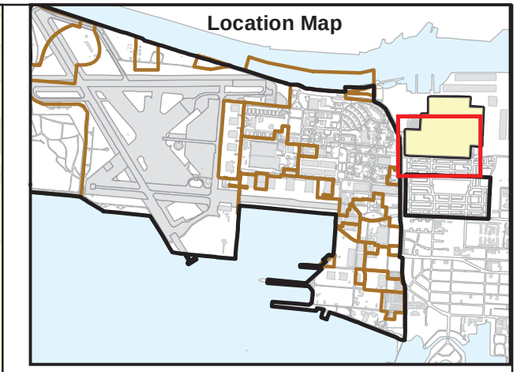
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 5
IR SITE 30

FIGURE
A-19





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 31 Installation Restoration Site and Number
- Operable Unit 5
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 200 400 Feet

N

PROJECT NO.:	5023-14-6096
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CHECKED BY:	KO

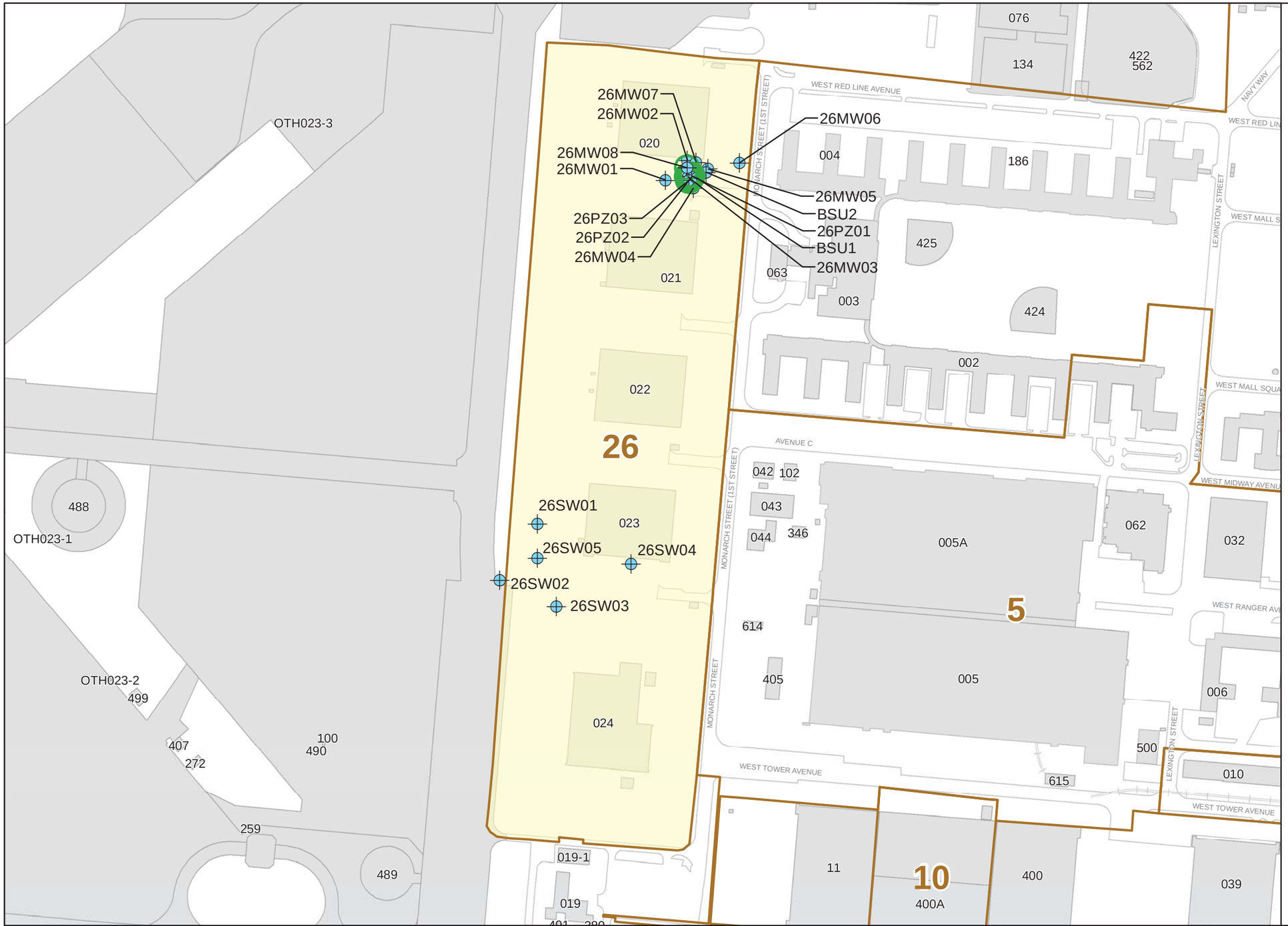
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

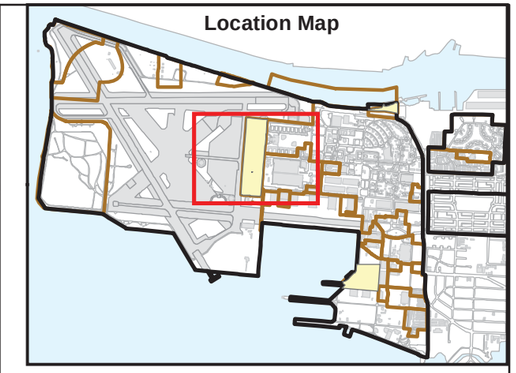
OU - 5
IR SITE 31

FIGURE

A-20



Location Map



Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 26 Installation Restoration Site and Number
- Operable Unit 6
- Facility Infrastructure

Notes:
Well 26MW09 is not shown because the survey coordinates are unknown.

All known and possibly existing groundwater monitoring wells and piezometers are shown (except as noted above), based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 300 600 Feet

N

PROJECT NO.:	5023-14-6096
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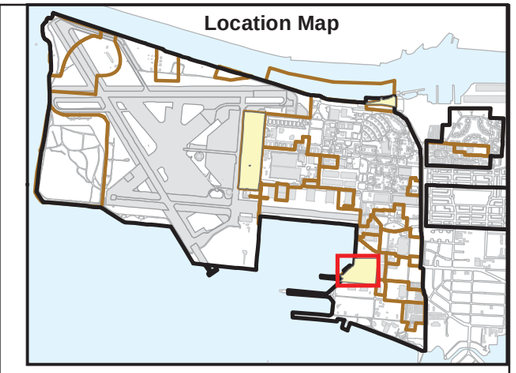
MNEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 6
IR SITE 26

FIGURE
A-21



Legend

- Groundwater Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- 27 Installation Restoration Site and Number
- Operable Unit 6
- Facility Infrastructure

Notes:
All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

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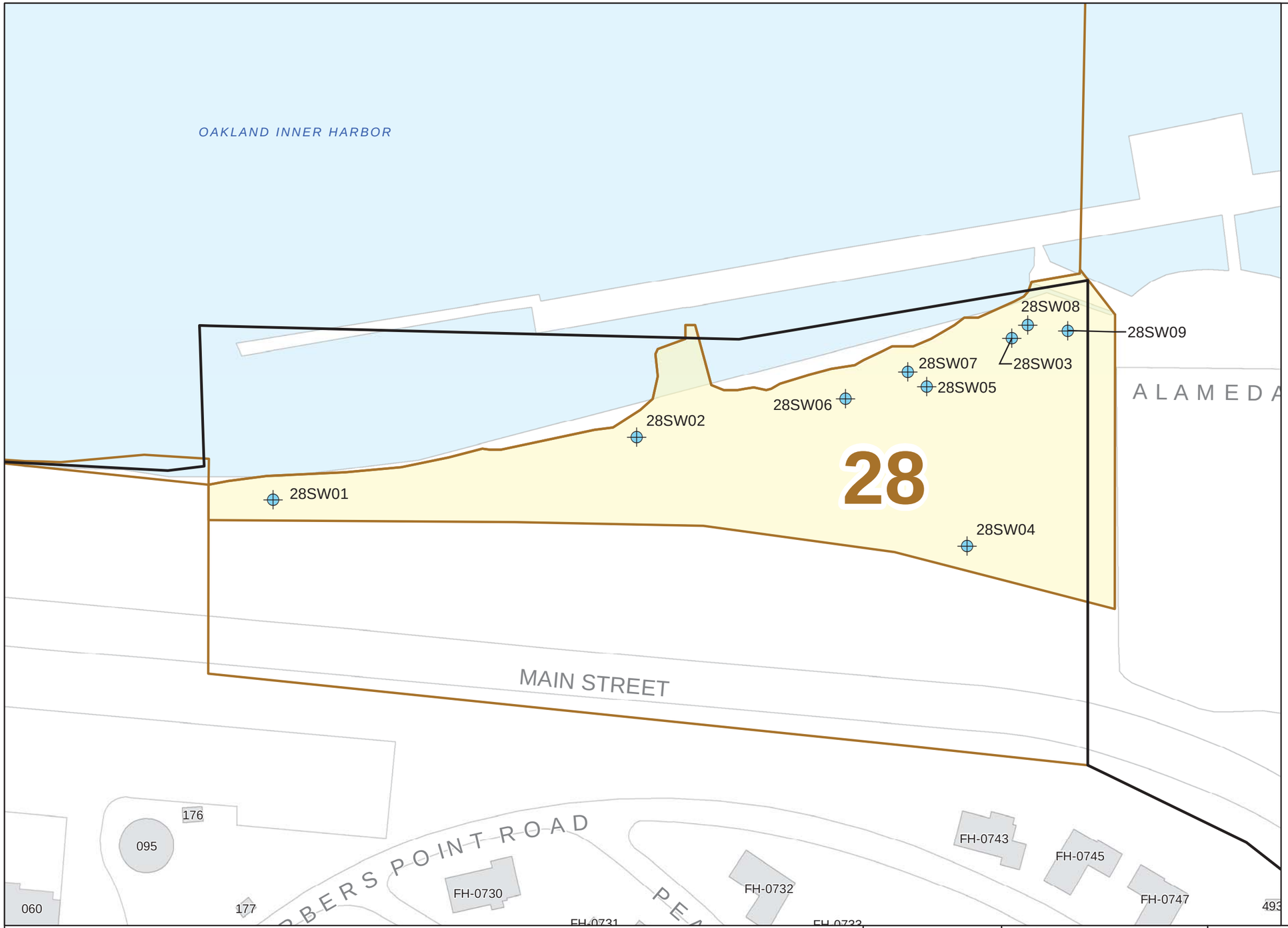
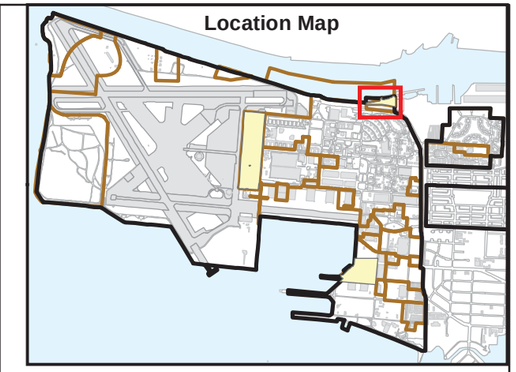
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 6
IR SITE 27

FIGURE
A-22





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 28 Installation Restoration Site and Number
- Operable Unit 6
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

PROJECT NO.:	5023-14-6096
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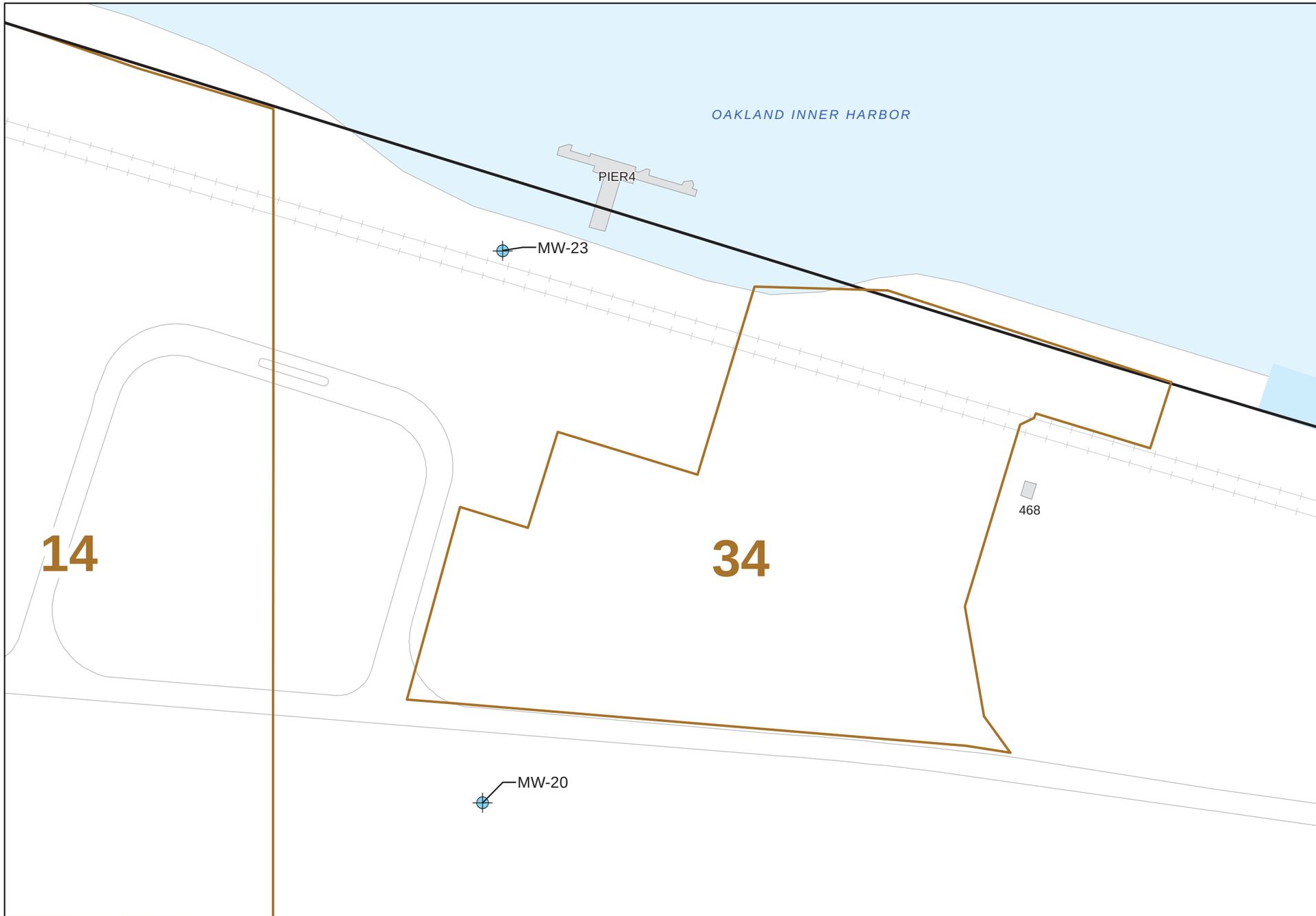
DRAWN BY:	SB
CHECKED BY:	KO

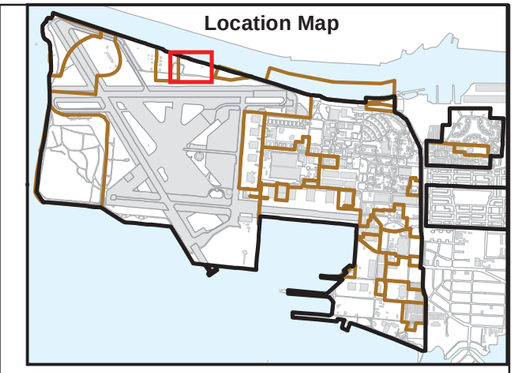
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OU - 6
IR SITE 28

FIGURE
A-23





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 34 Installation Restoration Site and Number
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 100 200 Feet

N

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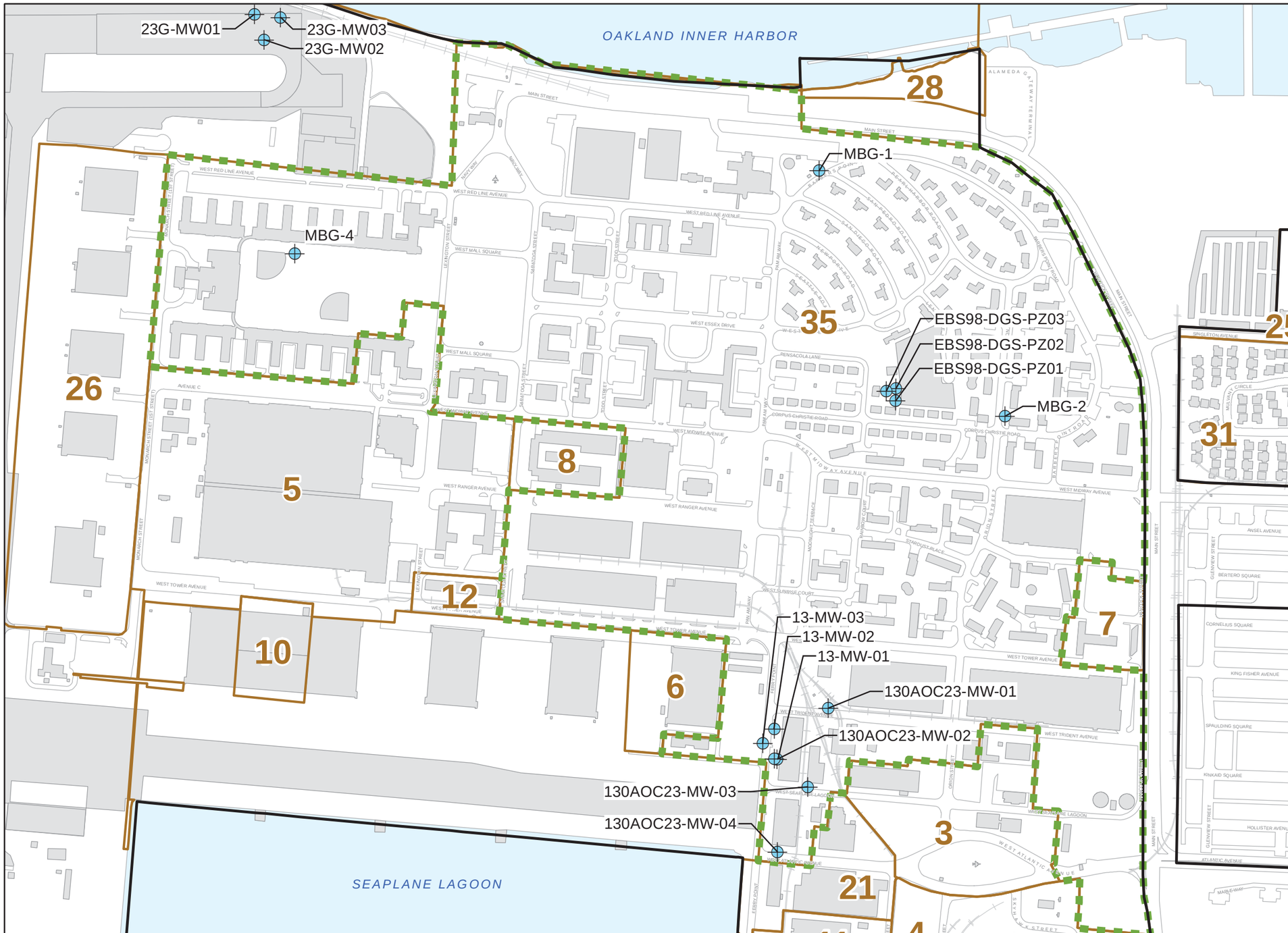
DRAWN BY:	SB
CHECKED BY:	KO

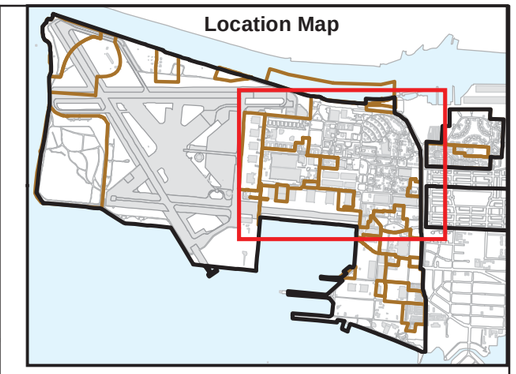
MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

IR SITE 34

FIGURE A-24





Legend

- Groundwater Monitoring Well
- Alameda Point Boundary
- 35 Installation Restoration Site and Number
- IR Site 35 Boundary
- Facility Infrastructure

Notes:
No wells at this site were sampled in 2014.

All known and possibly existing groundwater monitoring wells and piezometers are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes reports and databases for groundwater monitoring conducted in 2013 and 2014 (SES-TECH, 2014; SES-TECH, 2015), and database information from 2007 for wells not monitored in 2013 and 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

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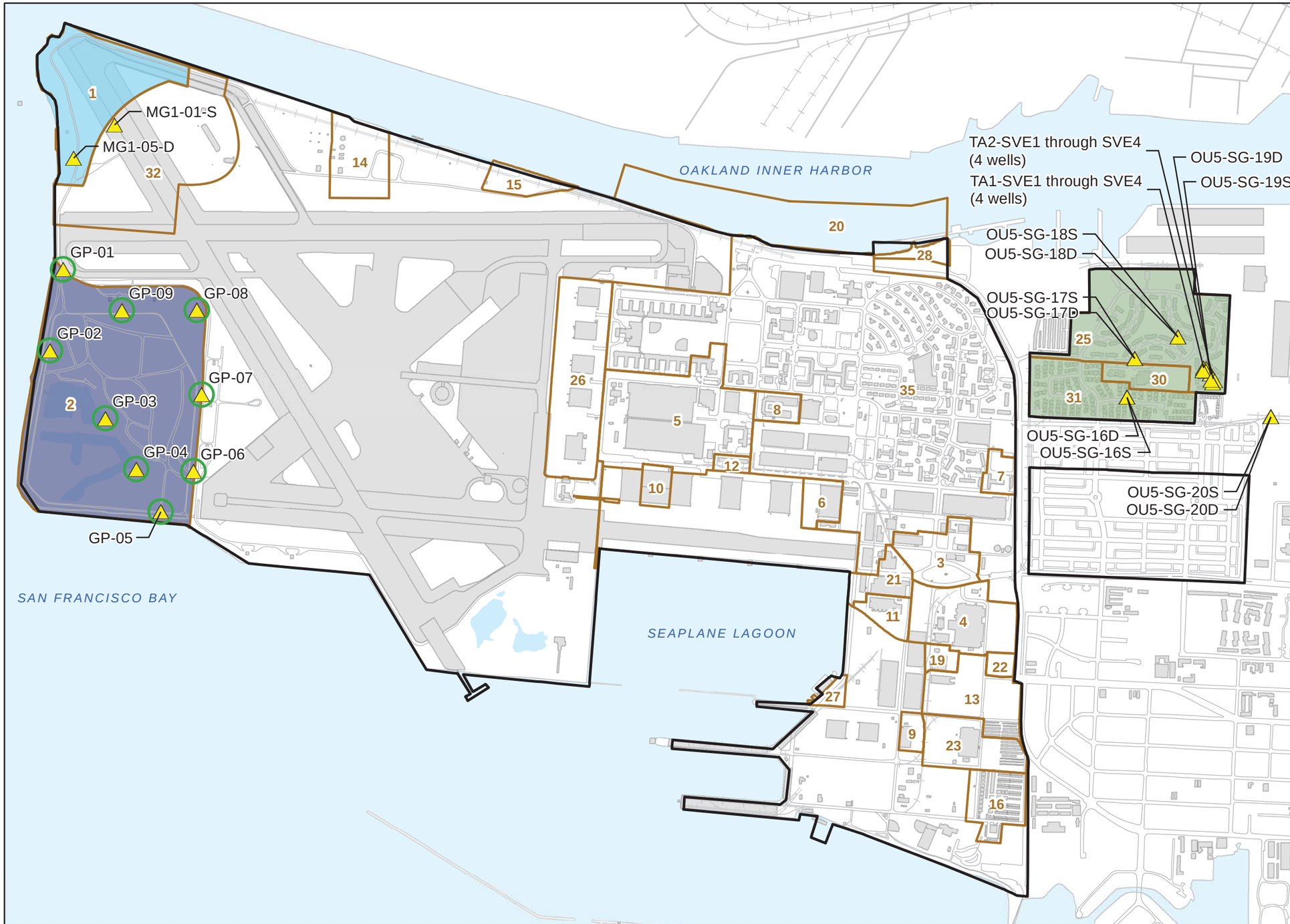
DATE: FEBRUARY 2015		IR SITE 35	A-25
DRAWN BY: SB			
CHECKED BY: KO			



Legend

- Soil Gas Monitoring Well
- Well Sampled in 2014
- Alameda Point Boundary
- Installation Restoration Site and Number
- Operable Unit 3
- Operable Unit 4A
- Operable Unit 5
- Facility Infrastructure

Notes:
All known and possibly existing soil gas monitoring wells are shown, based on a compilation of available well information provided by the Navy and SES-TECH. Well source information includes the report for monitoring conducted in 2014 (SES-TECH, 2015), and database information from 2007 for wells not monitored in 2014. Only selected wells will be sampled in accordance with the Basewide Groundwater Monitoring Program.

0 1,200 2,400 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
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MMEC GROUP

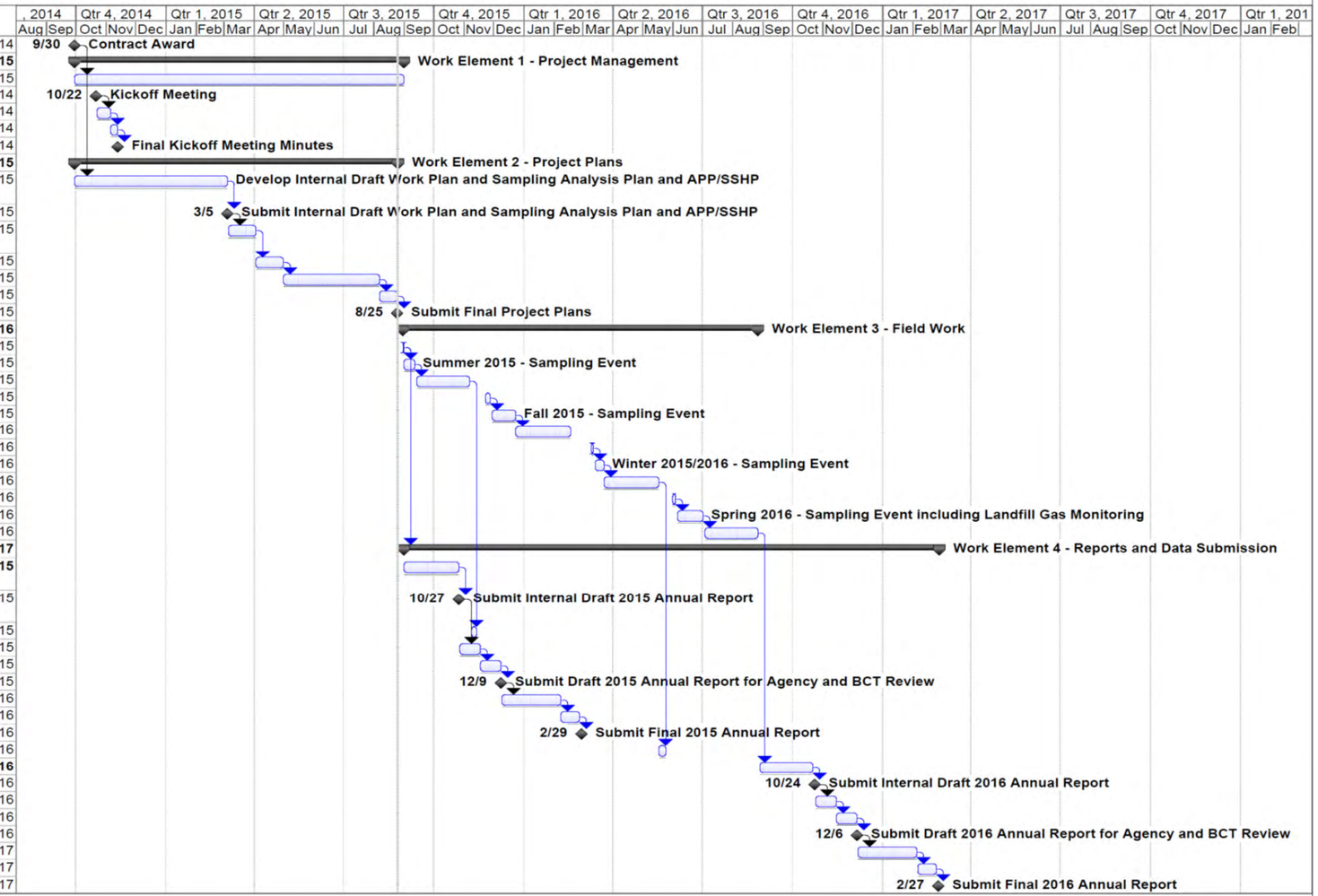
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

SOIL GAS MONITORING LOCATIONS

FIGURE A-26

Proposed Project Schedule
Installation Restoration Sites
Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California
N62473-12-D-2012 Task Order 0096

ID	Task Name	Duration	Start	Finish
1	Contract Award	0 days	Tue 9/30/14	Tue 9/30/14
2	**Work Element 1 - Project Management**	**240 days**	**Tue 9/30/14**	**Mon 8/31/15**
3	Project Management and Support	12 mons	Tue 9/30/14	Mon 8/31/15
4	Kickoff Meeting	1 day	Wed 10/22/14	Wed 10/22/14
5	Draft Kickoff Meeting Minutes	10 days	Thu 10/23/14	Wed 11/5/14
6	Navy Review - Draft Kickoff Meeting Minutes	5 days	Thu 11/6/14	Wed 11/12/14
7	Final Kickoff Meeting Minutes	3 days	Thu 11/13/14	Mon 11/17/14
8	**Work Element 2 - Project Plans**	**236 days**	**Tue 9/30/14**	**Tue 8/25/15**
9	Develop Internal Draft Work Plan and Sampling Analysis Plan and APP/SSHP	112 days	Tue 9/30/14	Wed 3/4/15
10	Submit Internal Draft Work Plan and Sampling Analysis Plan	1 day	Thu 3/5/15	Thu 3/5/15
11	Navy Review of Internal Draft Project Plans	20 days	Fri 3/6/15	Thu 4/2/15
12	Develop Draft Project Plans	20 days	Fri 4/3/15	Thu 4/30/15
13	Submit Draft Project Plans for Navy Review	70 days	Fri 5/1/15	Thu 8/6/15
14	Develop Final Project Plans	12 days	Fri 8/7/15	Mon 8/24/15
15	Submit Final Project Plans	1 day	Tue 8/25/15	Tue 8/25/15
16	**Work Element 3 - Field Work**	**260 days**	**Mon 8/31/15**	**Fri 8/26/16**
17	Summer 2015 - Mobilization	1 day	Mon 8/31/15	Mon 8/31/15
18	Summer 2015 - Sampling Event	9 days	Tue 9/1/15	Fri 9/11/15
19	Summer 2015 - Data Management	40 days	Mon 9/14/15	Fri 11/6/15
20	Fall 2015 - Mobilization	5 days	Mon 11/23/15	Fri 11/27/15
21	Fall 2015 - Sampling Event	18 days	Mon 11/30/15	Wed 12/23/15
22	Fall 2015 - Data Management	40 days	Thu 12/24/15	Wed 2/17/16
23	Winter 2015/2016 - Mobilization	2 days	Thu 3/10/16	Fri 3/11/16
24	Winter 2015/2016 - Sampling Event	7 days	Mon 3/14/16	Tue 3/22/16
25	Winter 2015/2016 - Data Management	40 days	Wed 3/23/16	Tue 5/17/16
26	Spring 2016 - Mobilization	3 days	Wed 6/1/16	Fri 6/3/16
27	Spring 2016 - Sampling Event including Landfill Gas Monitori	20 days	Mon 6/6/16	Fri 7/1/16
28	Spring 2016 - Data Management	40 days	Mon 7/4/16	Fri 8/26/16
29	**Work Element 4 - Reports and Data Submission**	**390 days**	**Tue 9/1/15**	**Mon 2/27/17**
30	**Develop Internal Draft 2015 Annual Report (begins after receipt of validated data from Spring 2015 event)**	**40 days**	**Tue 9/1/15**	**Mon 10/26/15**
31	Submit Internal Draft 2015 Annual Report	1 day	Tue 10/27/15	Tue 10/27/15
32	NIRIS Submittal - Spring and Summer 2015 Data	5 days	Mon 11/9/15	Fri 11/13/15
33	Navy Review of Internal Draft 2015 Annual Report	15 days	Wed 10/28/15	Tue 11/17/15
34	Develop Draft 2015 Annual Report	15 days	Wed 11/18/15	Tue 12/8/15
35	Submit Draft 2015 Annual Report for Agency and BCT Revie	1 day	Wed 12/9/15	Wed 12/9/15
36	Regulatory Agency and BCT Review - Draft 2015 Annual Re	60 edays	Wed 12/9/15	Sun 2/7/16
37	Develop Final 2015 Annual Report	15 days	Mon 2/8/16	Fri 2/26/16
38	Submit Final 2015 Annual Report	1 day	Mon 2/29/16	Mon 2/29/16
39	NIRIS Submittal - Fall 2015 and Winter 2015/2016 Data	5 days	Wed 5/18/16	Tue 5/24/16
40	**Develop Internal Draft 2016 Annual Report**	**40 days**	**Mon 8/29/16**	**Fri 10/21/16**
41	Submit Internal Draft 2016 Annual Report	1 day	Mon 10/24/16	Mon 10/24/16
42	Navy Review of Internal Draft 2016 Annual Report	15 days	Tue 10/25/16	Mon 11/14/16
43	Develop Draft 2016 Annual Report	15 days	Tue 11/15/16	Mon 12/5/16
44	Submit Draft 2016 Annual Report for Agency and BCT Revie	1 day	Tue 12/6/16	Tue 12/6/16
45	Regulatory Agency and BCT Review - Draft 2016 Annual Re	60 edays	Tue 12/6/16	Sat 2/4/17
46	Develop Final 2016 Annual Report	15 days	Mon 2/6/17	Fri 2/24/17
47	Submit Final 2016 Annual Report	1 day	Mon 2/27/17	Mon 2/27/17



Project:
Date: Tue 8/25/15

Task | Split | Progress | Milestone | Summary | Project Summary | External Tasks | External Milestone | Deadline

PROJECT NO.: 5023-14-6096	MMEC GROUP	Basewide Groundwater Monitoring Program Alameda Point, Alameda, California	FIGURE
DATE: August 2015		Project Schedule	A-27
DRAWN BY: SWB			
CHECKED BY: KO			

Attachment 1: Field Forms

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DAILY FIELD REPORT
PROJECT: **PROJECT No:**
DATE:
WEATHER: ° F
SUBCONTRACTOR (S) ON SITE :
EQUIPMENT ON SITE:
TASK PERFORMED BY AMEC AND SUBCONTRACTORS •
PROJECT OVERSIGHT AND QUALITY CONTROL MEASURES •
HEALTH AND SAFETY PROJECT ACTIVITIES: •
PROBLEMS ENCOUNTERED/CORRECTIVE MEASURES •
PROJECT NOTES (none, if any, describe): •
TOMORROW'S PLANNED ACTIVITIES: •
PREPARED BY:
REVIEWED BY:

CC:

File:

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MMEC GROUP	MMEC Group GROUNDWATER SAMPLING FIELD FORM	Job #: Task Order 0096 Date: Page of

Field Personnel: **Monitoring Well ID:**

Start Time: **Weather Conditions:** **Approx. Air Temp (F):**

INITIAL WELL DATA & WELL PURGING INFORMATION

Water Temperature (degree C)	Water pH (S.U.)	Specific Conductivity (µS/cm)	Turbidity (NTUs)	Dissolved Oxygen (mg/L)	ORP (mV)	Water Level (feet bgs)	Time (0:00 - 23:59)	Volume Purged (milliliters)
Stabilization:	3 consecutive readings within:							
±3%	±0.1 S.U.	±3%	±10% (if <5)	±10% (if <10)	±10 mV	-	-	-

Results of Field Ferrous Iron Kit (mg/L): **Total Purged:**

Measurements: 0.5 inch tubing: 0.020 gallons/linear foot 2" well casing: 0.17 gal/linear foot 4" well casing: 0.65 gal/linear foot

Purge Pumping Rate (approx. mL/min):

Approx. Pump/Intake Depth: screen

Well Yield: High / Moderate / Low Decontamination Method:

WELL CONDITION

Casing Size and Type:

Casing Condition:	OK / NA / Needs Repairs/Repaired	Lock Condition:	OK / NA / Needs Repairs/Repaired
Cap Condition:	OK / NA / Needs Repairs/Repaired	Monument Condition:	OK / NA / Needs Repairs/Repaired

NOTES:

SAMPLING INFORMATION / DATA

QA/QC Sample (circle one): Duplicate Lab QA/QC NONE

Sampling Method (circle one): dedicated Dual Valve Pump peristaltic pump

Analytical Parameters	Minimum Sample Vol.	Preservative	Bottle size	Number of bottles	Sample ID	Time Sampled

Method of Transportation of samples:

All samples were immediately placed into a cooler and packed with ice or "Blue Ice" YES / NO

Field Observations/Notes of Sampling Event:

Signature of Field Personnel:

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SHIP TO:

MMEC Group
9177 Sky Park Court
San Diego, CA 92123
(858) 514-6400

CHAIN OF CUSTODY

DATE: ______________

COC #: ______________

PAGE: ______ **OF** ______

Project Name:	Project Contact:	Bill To: AMEC	Disposal Instructions: LAB
Project Number:	Phone Number:		Shipment Method: COURIER/FedEx
Project Manager:	Project Phase:		Waybill Number: N/A

Sample Information							Methods for Analysis	RUSH						
No.	Sample ID	Date Sampled	Time Sampled	Matrix				24 Hour	48 Hour	72 Hour	5 Days	QC (MS/MSD)	TOTAL BOTTLES	HOLD All Analyses
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														
11														
12														

Sampler's Signature:	**Date:**	**Time:**
Relinquished By/Affiliation:	**Date:**	**Time:**
Received By:	**Date:**	**Time:**
Relinquished By/Affiliation:	**Date:**	**Time:**
Received By:	**Date:**	**Time:**
Relinquished By/Affiliation:	**Date:**	**Time:**
Received By (LAB):	**Date:**	**Time:**

For Lab Use

Does COC match samples: Y or N
Broken Container: Y or N
COC seal intact: Y or N
Other problems: Y or N
AMEC contacted: Y or N
Date contacted: ______________

Cooler Temperature at receipt: _______ C

Comments: **H=Hold Analysis Request X=Analyze**

NUMBER OF COOLERS SENT: ___

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Attachment 2: Laboratory Certifications

To be included with the Final SAP on CD

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CALIFORNIA
Water Boards
STATE WATER RESOURCES CONTROL BOARD
REGIONAL WATER QUALITY CONTROL BOARDS

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

CERTIFICATE OF ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

TestAmerica Denver

4955 Yarrow Street

Arvada, CO 80002

Scope of the certificate is limited to the
"Fields of Testing"
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site, proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **2513**

Expiration Date: **08/31/2016**

Effective Date: **09/01/2014**

Richmond, California
subject to forfeiture or revocation

Christine Sotelo, Chief
California State Environmental Laboratory Accreditation Program

California Environmental Protection Agency

State Water Resources Control Board

Environmental Laboratory Accreditation Program

EDMUND G. BROWN JR.
Governor

August 28, 2014

William S. Cicero
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Dear William S. Cicero: Certificate No. 2513

This is to advise you that the laboratory named above continues to be certified as an environmental testing laboratory pursuant to the provisions of the Health and Safety Code (HSC), Division 101, Part 1, Chapter 4, Section 100825, et seq. Certification for all currently certified Fields of Testing that the laboratory has applied for renewal shall remain in effect until **08/31/2016** unless it is revoked.

Please note that the renewal application for certification is subject to an on-site process, and the continued use of this certificate is contingent upon:

* **successful completion of the on-site process;**
* **acceptable performance in the required proficiency testing (PT) studies;**
* **timely payment of all fees, including an annual fee due before August 31, 2015;**
* **compliance with Environmental Laboratory Accreditation Program (ELAP); statutes (HSC, Section 100825, et seq.) and Regulations (California Code of Regulations (CCR),Title 22, Division 4, Chapter 19).**

An updated certificate of the "Fields of Testing" will be issued to the laboratory upon successful completion of the on-site process.

The application for the renewal of this certificate must be received before the expiration date to remain in force according to the HSC100845(a).

Please note that the laboratory is required to notify ELAP of any major changes in the laboratory such as the transfer of ownership, change of laboratory director, change in location, or structural alterations which may affect adversely the quality of analyses (HSC, Section 100845(b)(d)). Please include the above certificate number in all your correspondence with ELAP.

If you have any questions, please contact ELAP at (510) 620-3155.

Sincerely,

Christine Sotelo, Chief
California State Environmental Laboratory Accreditation Program

Environmental Laboratory Accreditation Program
850 Marina Bay Parkway, MS 0511, Bldg P, 1st Fl, Richmond, CA 94804
(510) 620-3155, FAX (510) 620-3165
www.cdph.ca.gov/certlic/labs/Pages/ELAP.aspx

CALIFORNIA DEPARTMENT OF PUBLIC HEALTH
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing

TestAmerica Laboratories, Inc. Denver
4955 Yarrow Street
Arvada, CO 80002
Phone: (303) 736-0100

Certificate No.: 2513
Renew Date: 8/31/2014

Field of Testing: 104 - Volatile Organic Chemistry of Drinking Water

104.030	001	1,2-Dibromoethane	EPA 504.1
104.030	002	1,2-Dibromo-3-chloropropane	EPA 504.1

Field of Testing: 108 - Inorganic Chemistry of Wastewater

108.090	001	Residue, Volatile	EPA 160.4
108.110	001	Turbidity	EPA 180.1
108.112	001	Boron	EPA 200.7
108.112	002	Calcium	EPA 200.7
108.112	003	Hardness (calculation)	EPA 200.7
108.112	004	Magnesium	EPA 200.7
108.112	005	Potassium	EPA 200.7
108.112	006	Silica	EPA 200.7
108.112	007	Sodium	EPA 200.7
108.120	001	Bromide	EPA 300.0
108.120	002	Chloride	EPA 300.0
108.120	003	Fluoride	EPA 300.0
108.120	004	Nitrate	EPA 300.0
108.120	005	Nitrite	EPA 300.0
108.120	006	Nitrate-nitrite	EPA 300.0
108.120	007	Phosphate, Ortho	EPA 300.0
108.120	008	Sulfate	EPA 300.0
108.183	001	Cyanide, Total	EPA 335.4
108.200	001	Ammonia	EPA 350.1
108.211	001	Kjeldahl Nitrogen	EPA 351.2
108.232	001	Nitrate-nitrite	EPA 353.2
108.232	002	Nitrite	EPA 353.2
108.260	001	Phosphate, Ortho	EPA 365.1
108.261	001	Phosphorus, Total	EPA 365.1
108.360	001	Phenols, Total	EPA 420.1
108.362	001	Phenols, Total	EPA 420.4
108.381	001	Oil and Grease	EPA 1664A
108.400	001	Acidity	SM2310B
108.410	001	Alkalinity	SM2320B
108.420	001	Hardness (calculation)	SM2340B

As of 10/4/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

108.430	001	Conductivity	SM2510B
108.440	001	Residue, Total	SM2540B
108.441	001	Residue, Filterable	SM2540C
108.442	001	Residue, Non-filterable	SM2540D
108.443	001	Residue, Settleable	SM2540F
108.470	001	Cyanide, Manual Distillation	SM4500-CN C
108.472	001	Cyanide, Total	SM4500-CN E
108.473	001	Cyanide, amenable	SM4500-CN G
108.490	001	pH	SM4500-H+ B
108.510	001	Nitrite	SM4500-NO2 B
108.560	001	Sulfite	SM4500-SO3 B
108.580	001	Sulfide	SM4500-S= D
108.582	001	Sulfide	SM4500-S= F (19th/20th)
108.590	001	Biochemical Oxygen Demand	SM5210B
108.591	001	Carbonaceous BOD	SM5210B
108.610	001	Total Organic Carbon	SM5310B-2000

Field of Testing: 109 - Toxic Chemical Elements of Wastewater

109.010	001	Aluminum	EPA 200.7
109.010	002	Antimony	EPA 200.7
109.010	003	Arsenic	EPA 200.7
109.010	004	Barium	EPA 200.7
109.010	005	Beryllium	EPA 200.7
109.010	007	Cadmium	EPA 200.7
109.010	009	Chromium	EPA 200.7
109.010	010	Cobalt	EPA 200.7
109.010	011	Copper	EPA 200.7
109.010	012	Iron	EPA 200.7
109.010	013	Lead	EPA 200.7
109.010	015	Manganese	EPA 200.7
109.010	016	Molybdenum	EPA 200.7
109.010	017	Nickel	EPA 200.7
109.010	019	Selenium	EPA 200.7
109.010	021	Silver	EPA 200.7
109.010	023	Thallium	EPA 200.7
109.010	024	Tin	EPA 200.7
109.010	026	Vanadium	EPA 200.7
109.010	027	Zinc	EPA 200.7
109.020	002	Antimony	EPA 200.8
109.020	003	Arsenic	EPA 200.8
109.020	004	Barium	EPA 200.8
109.020	005	Beryllium	EPA 200.8

109.020	006	Cadmium	EPA 200.8
109.020	007	Chromium	EPA 200.8
109.020	008	Cobalt	EPA 200.8
109.020	009	Copper	EPA 200.8
109.020	010	Lead	EPA 200.8
109.020	011	Manganese	EPA 200.8
109.020	012	Molybdenum	EPA 200.8
109.020	013	Nickel	EPA 200.8
109.020	014	Selenium	EPA 200.8
109.020	015	Silver	EPA 200.8
109.020	016	Thallium	EPA 200.8
109.020	017	Vanadium	EPA 200.8
109.020	018	Zinc	EPA 200.8
109.190	001	Mercury	EPA 245.1

Field of Testing: 110 - Volatile Organic Chemistry of Wastewater

110.020	000	Aromatic Volatiles	EPA 602
110.040	040	Halogenated Hydrocarbons	EPA 624
110.040	041	Aromatic Compounds	EPA 624
110.040	042	Oxygenates	EPA 624
110.040	043	Other Volatile Organics	EPA 624

Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater

111.060	000	Polynuclear Aromatics	EPA 610
111.101	032	Polynuclear Aromatic Hydrocarbons	EPA 625
111.101	033	Adipates	EPA 625
111.101	034	Phthalates	EPA 625
111.101	036	Other Extractables	EPA 625
111.120	999	N-nitrosodimethylamine	EPA 1625
111.170	030	Organochlorine Pesticides	EPA 608
111.170	031	PCBs	EPA 608

Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste

114.010	001	Antimony	EPA 6010B
114.010	002	Arsenic	EPA 6010B
114.010	003	Barium	EPA 6010B
114.010	004	Beryllium	EPA 6010B
114.010	005	Cadmium	EPA 6010B
114.010	006	Chromium	EPA 6010B
114.010	007	Cobalt	EPA 6010B
114.010	008	Copper	EPA 6010B
114.010	009	Lead	EPA 6010B
114.010	010	Molybdenum	EPA 6010B

114.010	011	Nickel	EPA 6010B	
114.010	012	Selenium	EPA 6010B	
114.010	013	Silver	EPA 6010B	
114.010	014	Thallium	EPA 6010B	
114.010	015	Vanadium	EPA 6010B	
114.010	016	Zinc	EPA 6010B	
114.020	001	Antimony	EPA 6020	
114.020	002	Arsenic	EPA 6020	
114.020	003	Barium	EPA 6020	
114.020	004	Beryllium	EPA 6020	
114.020	005	Cadmium	EPA 6020	
114.020	006	Chromium	EPA 6020	
114.020	007	Cobalt	EPA 6020	
114.020	008	Copper	EPA 6020	
114.020	009	Lead	EPA 6020	
114.020	010	Molybdenum	EPA 6020	
114.020	011	Nickel	EPA 6020	
114.020	012	Selenium	EPA 6020	
114.020	013	Silver	EPA 6020	
114.020	014	Thallium	EPA 6020	
114.020	015	Vanadium	EPA 6020	
114.020	016	Zinc	EPA 6020	
114.103	001	Chromium (VI)	EPA 7196A	
114.140	001	Mercury	EPA 7470A	
114.141	001	Mercury	EPA 7471A	
114.221	001	Cyanide, Total	EPA 9012A	
114.230	001	Sulfides, Total	EPA 9034	
114.240	001	Corrosivity - pH Determination	EPA 9040B	
114.241	001	Corrosivity - pH Determination	EPA 9045C	
114.250	001	Fluoride	EPA 9056	
114.280	001	Organic Lead	HML 939-M	Interim

Field of Testing: 115 - Extraction Test of Hazardous Waste

115.020	001	Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311	
115.030	001	Waste Extraction Test (WET)	CCR Chapter11, Article 5, Appendix II	
115.040	001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312	

Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste

116.010	000	EDB and DBCP	EPA 8011	
116.020	030	Nonhalogenated Volatiles	EPA 8015B	
116.020	031	Ethanol and Methanol	EPA 8015B	
116.030	001	Gasoline-range Organics	EPA 8015B	
116.040	041	Methyl tert-butyl Ether (MTBE)	EPA 8021B	

116.040	061	Aromatic Volatiles	EPA 8021B
116.040	062	BTEX	EPA 8021B
116.080	000	Volatile Organic Compounds	EPA 8260B
116.080	120	Oxygenates	EPA 8260B

Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.010	001	Diesel-range Total Petroleum Hydrocarbons	EPA 8015B
117.110	000	Extractable Organics	EPA 8270C
117.140	000	Polynuclear Aromatic Hydrocarbons	EPA 8310
117.170	000	Nitroaromatics and Nitramines	EPA 8330
117.210	000	Organochlorine Pesticides	EPA 8081A
117.220	000	PCBs	EPA 8082
117.240	000	Organophosphorus Pesticides	EPA 8141A
117.250	000	Chlorinated Herbicides	EPA 8151A
117.280	000	Carbamates	EPA 8321A

Field of Testing: 120 - Physical Properties of Hazardous Waste

120.010	001	Ignitability	EPA 1010
120.070	001	Corrosivity - pH Determination	EPA 9040B
120.080	001	Corrosivity - pH Determination	EPA 9045C

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SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA DENVER
4955 Yarrow Street
Arvada, CO 80002
Margaret S. Sleevi Phone: 303-736-0100
www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2015 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality Systems Manual for Environmental Laboratories), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Metals				
Aluminum	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Antimony	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Arsenic	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Barium	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Beryllium	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Boron	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Cadmium	EPA 6010C	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A

5301 Buckeystown Pike, Suite 350 | Frederick, Maryland 21704-8373 | Phone: 301 644 3248 | Fax: 301 662 2974 | www.A2LA.org

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Calcium	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Chromium	EPA 6010C	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Cobalt	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Copper	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Iron	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Lead	EPA 6010C	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Lithium	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Magnesium	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Manganese	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Mercury	----------	----------	EPA 7470A	EPA 7471A / 7471B
Molybdenum	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Nickel	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Potassium	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Selenium	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Silica	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Silicon	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Silver	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Sodium	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Strontium	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Thallium	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Tin	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Titanium	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Vanadium	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Zinc	----------	----------	EPA 6010B / 6010C / 6020 / 6020A	EPA 6010B / 6010C / 6020 / 6020A
Nutrients				
Nitrate (as N)	----------	By calculation	By calculation / EPA 9056 / 9056A	By calculation / EPA 9056 / 9056A
Nitrate-nitrite (as N)	----------	EPA 353.2	EPA 353.2 / 9056 / 9056A	EPA 9056 / 9056A
Nitrite (as N)	----------	SM 4500-NO2 B	SM 4500-NO2 B; EPA 9056 / 9056A	EPA 9056 / 9056A
Orthophosphate (as P)	----------	----------	EPA 9056 / 9056A	EPA 9056 / 9056A
Total phosphorus	----------	----------	EPA 6010B / 6010C	EPA 6010B / 6010C
Demands				
Total Organic Carbon	----------	----------	EPA 9060 / 9060A	EPA 9060 / 9060A
Total Organic Halides	----------	----------	EPA 9020B	----------
Wet Chemistry				----------
Alkalinity (Total Bicarbonate, Carbonate, and Hydroxide Alkalinty)	----------	SM 2320 B_1997	SM 2320 B	SM 2320 B
Ammonia	----------	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	----------	SM 5210B	SM 5210B	----------
Bromide	----------	----------	EPA 9056 / 9056A	EPA 9056 / 9056A
Chloride	----------	----------	EPA 9056 / 9056A	EPA 9056 / 9056A
Chemical Oxygen Demand	----------	EPA 410.4	EPA 410.4	
Conductivity	----------	----------	EPA 9050 / 9050A	EPA 9050 / 9050A
Cyanide	----------	----------	9012A / 9012B	9012A / 9012B
Ferrous Iron	----------	SM 3500 Fe B, D	SM 3500 Fe B, D	----------
Fluoride	----------	----------	EPA 9056 / 9056A	EPA 9056 / 9056A
Hexavalent Chromium	EPA 7196A	----------	EPA 7196A	----------
pH	----------	----------	EPA 9040B / 9045C	EPA 9040B / 9045C
Oil and Grease (HEM and SGT-HEM)	----------	----------	EPA 1664A/ 1664B	9071B
Percent Moisture	----------	----------	----------	ASTM D2216
Perchlorate	----------	----------	EPA 6860	EPA 6860
Phenols	----------	----------	EPA 9066	EPA 9066
Solids, Total	----------	SM 2540 B	SM 2540 B	SM 2540 B
Solids, Total Suspended	----------	SM 2540 D	SM 2540 D	SM 2540 D
Solids, Total Dissolved	----------	SM 2540 C	SM 2540 C	SM 2540 C
Sulfate	----------	----------	EPA 9056 / 9056A	EPA 9056 / 9056A
Sulfide, Total	----------	----------	EPA 9034	EPA 9034
Sulfide	----------	----------	EPA 9030B	EPA 9030B
Total Kjeldahl Nitrogen	----------	----------	EPA 351.2	----------

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Purgeable Organics (volatiles)				----------
Acetone	----------	----------	EPA 8260B	EPA 8260B
Acetonitrile	----------	----------	EPA 8260B	EPA 8260B
Acrolein	----------	----------	EPA 8260B	EPA 8260B
Acrylonitrile	----------	----------	EPA 8260B	EPA 8260B
Allyl Chloride	----------	----------	EPA 8260B	EPA 8260B
tert-Amyl Methyl Ether	EPA 8260B			
Benzene	EPA 8260B / 8021B	----------	EPA 8260B / 8021B / AK101/ OK DEQ GRO/8260B SIM	EPA 8260B / 8021B / AK101/ OK DEQ GRO
Bromobenzene	----------	----------	EPA 8260B	EPA 8260B
Bromochloromethane	----------	----------	EPA 8260B	EPA 8260B
Bromodichloromethane	----------	----------	EPA 8260B	EPA 8260B
Bromoform	----------	----------	EPA 8260B	EPA 8260B
Bromomethane	----------	----------	EPA 8260B	EPA 8260B
2-Butanone	----------	----------	EPA 8260B	EPA 8260B
n-Butyl alcohol	----------	----------	EPA 8260B / 8015B / 8015C	EPA 8260B / 8015B / 8015C
tert-Butyl alcohol (2-Methyl-2-Propanol)	EPA 8260B		EPA 8260B/ 8260B SIM	EPA 8260B
n-Butylbenzene	----------	----------	EPA 8260B	EPA 8260B
sec-Butylbenzene	----------	----------	EPA 8260B	EPA 8260B
tert-Butylbenzene	----------	----------	EPA 8260B	EPA 8260B
Carbon disulfide	----------	----------	EPA 8260B	EPA 8260B
Carbon tetrachloride	----------	----------	EPA 8260B	EPA 8260B
Chlorobenzene	----------	----------	EPA 8260B / 8021B	EPA 8260B / 8021B
2-Chloro-1,3-butadiene	----------	----------	EPA 8260B	EPA 8260B
Chloroethane	----------	----------	EPA 8260B	EPA 8260B
2-Chloroethyl vinyl ether	----------	----------	EPA 8260B	EPA 8260B
Chloroform	----------	----------	EPA 8260B	EPA 8260B
1-Chlorohexane	----------	----------	EPA 8260B	EPA 8260B
Chloromethane	----------	----------	EPA 8260B	EPA 8260B
Chloroprene	----------	----------	EPA 8260B	EPA 8260B
4-Chlorotoluene	----------	----------	EPA 8260B	EPA 8260B
2-Chlorotoluene	----------	----------	EPA 8260B	EPA 8260B
Cyclohexane	----------	----------	EPA 8260B	EPA 8260B
Cyclohexanone	----------	----------	EPA 8260B	EPA 8260B
Dibromochloromethane	----------	----------	EPA 8260B	EPA 8260B
1,2-Dibromo-3-chloropropane (DBCP)	----------	EPA 504	EPA 504 / 8260B / 8011	EPA 8260B / 8011
Dibromochloromethane	----------	----------	EPA 8260B	EPA 8260B
Dichlorodifluoromethane	----------	----------	EPA 8260B	EPA 8260B
Dibromomethane	----------	----------	EPA 8260B	EPA 8260B
1,2 Dibromoethane (EDB)	EPA 8011	EPA 504	EPA 504 / 8260B / 8011	EPA 8260B / 8011
1,2-Dichlorobenzene	----------	----------	EPA 8260B / 8021B	EPA 8260B / 8021B

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1,3-Dichlorobenzene	----------	----------	EPA 8260B / 8021B	EPA 8260B / 8021B
1,4-Dichlorobenzene	----------	----------	EPA 8260B / 8021B	EPA 8260B / 8021B
cis-1,4-Dichloro-2-butene	----------	----------	EPA 8260B	EPA 8260B
trans-1,4-Dichloro-2-butene	----------	----------	EPA 8260B	EPA 8260B
1,1-Dichloroethane	----------	----------	EPA 8260B	EPA 8260B
1,2-Dichloroethane	EPA 8260B	----------	EPA 8260B	EPA 8260B
1,1-Dichloroethene	----------	----------	EPA 8260B	EPA 8260B
1,2-Dichloroethene	----------	----------	EPA 8260B	EPA 8260B
cis-1,2-Dichloroethene	----------	----------	EPA 8260B	EPA 8260B
trans-1,2-Dichloroethene	----------	----------	EPA 8260B	EPA 8260B
Dichlorofluoromethane	----------	----------	EPA 8260B	EPA 8260B
1,2-Dichloropropane	----------	----------	EPA 8260B	EPA 8260B
1,3-Dichloropropane	----------	----------	EPA 8260B	EPA 8260B
2,2-Dichloropropane	----------	----------	EPA 8260B	EPA 8260B
1,1-Dichloropropene	----------	----------	EPA 8260B	EPA 8260B
1,3-Dichloropropene	----------	----------	EPA 8260B	EPA 8260B
cis-1,3-Dichloropropene	----------	----------	EPA 8260B	EPA 8260B
trans-1,3-Dichloropropene	----------	----------	EPA 8260B	EPA 8260B
Diethyl ether	----------	----------	EPA 8260B	EPA 8260B
Di-isopropylether	EPA 8260B	----------	EPA 8260B	EPA 8260B
1,4-Dioxane	----------	----------	EPA 8260B / 8260B SIM	EPA 8260B / 8260B SIM
Ethanol	----------	----------	EPA 8260B / 8015B / 8015C	EPA 8260B / 8015B / 8015C
Ethyl Acetate	----------	----------	EPA 8260B	EPA 8260B
Ethyl Benzene	EPA 8260B/8021B	----------	EPA 8260B / 8021B / AK101/ OK DEQ GRO	EPA 8260B / 8021B/ AK101/ OK DEQ GRO
Ethyl Methacrylate	----------	----------	EPA 8260B	EPA 8260B
Ethyl tert-Butyl Ether	EPA 8260B			
Ethylene Glycol	----------	----------	EPA 8015C	EPA 8015C
Gas Range Organics (GRO)	EPA 8015C	----------	EPA 8015B / 8015C / AK101 / 8015D	EPA 8015B / 8015C / AK101 / 8015D
Hexane	----------	----------	EPA 8260B	EPA 8260B
2-Hexanone	----------	----------	EPA 8260B	EPA 8260B
Hexachlorobutadiene	----------	----------	EPA 8260B	EPA 8260B
Isobutyl Alcohol (2-Methyl-1-propanol)	----------	----------	EPA 8260B / 8015B / 8015C	EPA 8260B / 8015B / 8015C
Isopropyl Alcohol	----------	----------	EPA 8260B	EPA 8260B
Isopropylbenzene	----------	----------	EPA 8260B	EPA 8260B
1,4-Isopropyltoluene	----------	----------	EPA 8260B	EPA 8260B
Iodomethane	----------	----------	EPA 8260B	EPA 8260B
Methacrylonitrile	----------	----------	EPA 8260B	EPA 8260B
Methanol	----------	----------	EPA 8015B / 8015C	EPA 8015B / 8015C
Methyl Acetate	----------	----------	EPA 8260B	EPA 8260B
Methyl Cyclohexane	----------	----------	EPA 8260B	EPA 8260B
Methylene Chloride	----------	----------	EPA 8260B	EPA 8260B
Methyl Ethyl Ketone (MEK)	----------	----------	EPA 8260B	EPA 8260B

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Methyl Isobutyl Ketone	----------	----------	EPA 8260B	EPA 8260B
Methyl Methacrylate	----------	----------	EPA 8260B	EPA 8260B
Methyl tert-Butyl Ether (MtBE)	EPA 8260B / 8021B	----------	EPA 8260B / 8021B / OK DEQ GRO	EPA 8260B / 8021B/ OK DEQ GRO
4-Methyl-2-Pentanone	----------	----------	EPA 8260B	EPA 8260B
Naphthalene	EPA 8260B / 8021B	----------	EPA 8260B/ OK DEQ GRO	EPA 8260B / OK DEQ GRO
2-Nitropropane	----------	----------	EPA 8260B	EPA 8260B
2,2' Oxybisethanol	----------	----------	EPA 8015C	EPA 8015C
2-Pentanone	----------	----------	EPA 8260B	EPA 8260B
Propionitrile	----------	----------	EPA 8260B	EPA 8260B
n-Propylbenzene	----------	----------	EPA 8260B	EPA 8260B
Propylene Glycol	----------	----------	EPA 8015C	EPA 8015C
Styrene	----------	----------	EPA 8260B	EPA 8260B
1,1,1,2-Tetrachloroethane	----------	----------	EPA 8260B	EPA 8260B
1,1,2,2-Tetrachloroethane	----------	----------	EPA 8260B	EPA 8260B
Tetrachloroethene	----------	----------	EPA 8260B	EPA 8260B
Tetrahydrofuran	----------	----------	EPA 8260B	EPA 8260B
Toluene	EPA 8260B / 8021B	----------	EPA 8260B / 8021B / AK101 / OK DEQ GRO	EPA 8260B / 8021B / AK101 / OK DEQ GRO
Total Petroleum Hydrocarbons (TPH)	----------	EPA 1664A EPA 1664B	EPA 1664A EPA 1664B	----------
1,2,3-Trichlorobenzene	----------	----------	EPA 8260B	EPA 8260B
1,1,1-Trichloroethane	----------	----------	EPA 8260B	EPA 8260B
1,1,2-Trichloroethane	----------	----------	EPA 8260B	EPA 8260B
Trichloroethene	----------	----------	EPA 8260B/ 8260B SIM	EPA 8260B
Trichlorofluoromethane	----------	----------	EPA 8260B	EPA 8260B
1,2,3-Trichlorobenzene	----------	----------	EPA 8260B	EPA 8260B
1,2,4-Trichlorobenzene	----------	----------	EPA 8260B	EPA 8260B
1,2,3-Trichloropropane	----------	EPA 504.1	EPA 504.1 / 8260B / 8011	EPA 8260B / 8011
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	----------	EPA 8260B	EPA 8260B
Triethylene Glycol	----------	----------	EPA 8015C	EPA 8015C
1,2,3-Trimethylbenzene	----------	----------	EPA 8260B	EPA 8260B
1,2,4-Trimethylbenzene	----------	----------	EPA 8260B	EPA 8260B
1,3,5-Trimethylbenzene	----------	----------	EPA 8260B	EPA 8260B
Vinyl Acetate	----------	----------	EPA 8260B	EPA 8260B
Vinyl Chloride	----------	----------	EPA 8260B/ 8260B SIM	EPA 8260B
Xylenes, total	EPA 8260B / 8021B	----------	EPA 8260B / 8021B / AK101 / OK DEQ GRO	EPA 8260B / 8021B / AK101 / OK DEQ GRO
1,2-Xylene	EPA 8260B / 8021B	----------	EPA 8260B / 8021B / AK101 / OK DEQ GRO	EPA 8260B / 8021B / AK101 / OK DEQ GRO
M+P-Xylene	EPA 8260B / 8021B	----------	EPA 8260B / 8021B / AK101 / OK DEQ GRO	EPA 8260B / 8021B / AK101 / OK DEQ GRO

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Methane	----------	----------	RSK-175	----------
Ethane	----------	----------	RSK-175	----------
Ethylene (Ethene)	----------	----------	RSK-175	----------
Acetylene	----------	----------	RSK-175	----------
Acetylene Ethane	----------	----------	RSK-175	----------
Extractable Organics (semivolatiles)				
Acenaphthene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Acenaphthylene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Acetophenone	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Acetylaminofluorene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Alachlor	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
4-Aminobiphenyl	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Aniline	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Anthracene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Aramite	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Atrazine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Azobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Benzaldehyde	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Benzidine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Benzoic acid	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Benzo (a) Anthracene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Benzo (b) Fluoranthene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Benzo (k) Fluoranthene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Benzo (ghi) Perylene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Benzo (a) Pyrene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Benzyl Alcohol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Bis (2-chloroethoxy) methane	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Bis (2-chloroethyl) Ether	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Bis (2-chloroisopropyl) Ether (2,2'Oxybis(1-chloropropane)	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Bis (2-ethylhexyl) Phthalate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
4-Bromophenyl Phenyl Ether	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Butyl Benzyl Phthalate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-sec-Butyl-4,6-Dinitrophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Carbazole	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
4-Chloroanilene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Chlorobenzilate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
4-Chloro-3-Methylphenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1-Chloronaphthalene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Chloronaphthalene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Chlorophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
4-Chlorophenyl Phenyl Ether	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Chrysene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Cresols	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Diallate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Dibenzo (a,h) Anthracene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Dibenzofuran	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,2-Dichlorobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,3-Dichlorobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,4-Dichlorobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
3,3'-Dichlorobenzidine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,4-Dichlorophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,6-Dichlorophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Diethyl phthalate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Dimethoate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
3,3-Dimethylbenzidine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
p-Dimethylaminoazobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
7,12-Dimethylbenz(a)anthracene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Alpha-,alpha-Dimethylphenethylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,4-Dimethylphenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Dimethyl Phthalate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Di-n-Butyl Phthalate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Di-n-Octyl Phthalate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,3-Dinitrobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,4-Dinitrobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,4-Dinitrophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,4-Dinitrotoluene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,6-Dinitrotoluene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,4-Dioxane	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Diphenylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,2-Diphenylhydrazine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Disulfoton	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Diesel Range Organics (DRO)	EPA 8015C	----------	EPA 8015B / 8015C, AK102, TX 1005 / 8015D / OK DEQ DRO	EPA 8015B / 8015C, AK102, TX 1005 / 8015D / OK DEQ DRO
Ethyl Methanesulfonate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Famphur	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Fluoroanthene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Fluorene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Gasoline Range Organics	----------	----------	TX 1005 / OK DEQ GRO	TX 1005 / OK DEQ GRO
Hexachlorobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Hexachlorobutadiene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Hexachlorocyclopentadiene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Hexachloroethane	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Hexachloropropene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Indeno (1,2,3-cd) Pyrene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Isodrin	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Isophorone	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Isosafrole	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Methapyrilene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
3-Methylcholanthrene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Methyl-4,6-Dinitrophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Methyl Methane Sulfonate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Methylcholanthrene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1-Methylnaphthalene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
2-Methylnaphthalene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
2-Methylphenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
3+4-Methylphenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Naphthalene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
1,4-Naphthoquinone	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1-Naphthylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Naphthylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Nitroaniline	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
3-Nitroaniline	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
4-Nitroaniline	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Nitrobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Nitrophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
4-Nitrophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Nitroquinoline-1-Oxide	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosodiethylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosodimethylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosodi-n-Butylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosodi-n-Propylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosodiphenylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosomethylethylamine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosomorpholine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosopiperidine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
N-Nitrosopyrrolidine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
5-Nitro-o-Toluidine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,2-oxybis(1-chloropropane)	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Parathion, Methyl	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Parathion, Ethyl	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Pentachlorobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Pentachloroethane	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Pentachloronitobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Pentachlorophenol	----------	----------	EPA 8270C / 8270D / 8321A / 8321B	EPA 8270C / 8270D / 8321A / 8321B
Phenacetin	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Phenanthrene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Phenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Phorate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2-Picoline	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Pronamide	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Pyrene	----------	----------	EPA 8270C / 8270D / 8270SIM	EPA 8270C / 8270D / 8270SIM
Pyridine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Safrole	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Sulfotepp	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,2,4,5-Tetrachlorobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,3,4,6-Tetrachlorophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Thionazin	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
o-Toluidine	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,2,4-Trichlorobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,4,5-Trichlorophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
2,4,6-Trichlorophenol	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
o,o,o-Triethyl Phosphorothioate	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
1,3,5-Trinitrobenzene	----------	----------	EPA 8270C / 8270D	EPA 8270C / 8270D
Motor Oil (Residual Range Organics)	----------	----------	EPA 8015B / 8015C, AK103 / OK DEQ RRO	EPA 8015B / 8015C, AK103 / OK DEQ RRO
Pesticides/Herbicides/PCBs				
Aldrin	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Atrazine	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Azinophos ethyl	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Azinophos methyl	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
alpha-BHC	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
beta-BHC	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
delta-BHC	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
gamma-BHC	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Bolstar	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
alpha-Chlordane	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
gamma-Chlordane	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Chlordane (technical)	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Chloropyrifos	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Coumaphos	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
2,4-D	----------	----------	EPA 8151A / 8321A	EPA 8151A /8321A
Dalapon	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
2,4-DB	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
4,4'-DDD	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
4,4'-DDE	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
4,4'-DDT	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Demeton-O	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Demeton-S	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Demeton, total	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Diazinon	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Dicamba	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
Dichlorovos	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Dichloroprop	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
Dieldrin	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Dimethoate	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Dinoseb	----------	----------	EPA 8151A / 8321A	EPA 8321A
Disulfoton	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Endosulfan I	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Endosulfan II	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Endonsulfan sulfate	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Endrin	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Endrin aldehyde	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Endrin ketone	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
EPN	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Ethoprop	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Ethyl Parathion	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Famphur	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Fensulfothion	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Fenthion	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Heptachlor	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Heptachlor Epoxide	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Hexachlorobenzene	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Malathion	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
MCPA	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
MCPP	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
Merphos	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Methoxychlor	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Methyl parathion	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Mevinphos	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Naled	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
PCB-1016 (Arochlor)	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1221	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1232	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1242	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1248	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1254	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1260	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1262	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
PCB-1268	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
Total PCBs	----------	----------	EPA 8082 / 8082A	EPA 8082 / 8082A
Phorate	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Phosmet	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Propazine	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Ronnel	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Simazine	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Stirophos	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Sulfotepp	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
2,4,5-T	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
Thionazin	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Tokuthion	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
2,4,5-TP	----------	----------	EPA 8151A / 8321A	EPA 8151A / 8321A
Toxaphene	----------	----------	EPA 8081A / 8081B	EPA 8081A / 8081B
Trichloronate	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
o,o,o-Triethylphos Phorothioate	----------	----------	EPA 8141A / 8141B	EPA 8141A / 8141B
Explosives				
1,3,5-Trinitrobenzene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
1,3-Dinitrobenzene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
2,4,6-Trinitrotoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
3,5-Dinitroaniline	----------	----------	EPA 8330B	EPA 8330B
2,4-Dinitrotoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
2,6-Dinitroltoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
2-Amino-4,6-Dinitrotoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
2-Nitrotoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
3-Nitrotoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
4-Amino-2,6-Dinitrotoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
4-Nitrotoluene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
Nitrobenzene	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
Nitroglycerin	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
Octahydro-1,3,5,7-Tetrabitro-1,3,5,7-Tetrazocine (HMX)	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
Pentaerythritoltetranitrate (PETN)	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
Picric acid	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
RDX (Hexahydro-1,3,5-Trinitro-1,3,5-Triazine)	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
Tetryl (Methyl 2,4,6-Trinitrophenylnitramine	----------	----------	EPA 8330A / 8330B / 8321A / 8321B	EPA 8330A / 8330B / 8321A / 8321B
Perfluorinated Hydrocarbons (PFCs) and Perfluorinated Sulfonates (PFSs)				
Perfluorobutanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoropentanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorohexanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoroheptanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorononanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorodecanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoroundecanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorododecanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorotridecanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorotetradecanoic Acid	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorobutane Sulfonate	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorohexane Sulfonate	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctane Sulfonate	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorodecane Sulfonate	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctane Sulfonamide	----------	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Hazardous Waste Characteristics				
Conductivity	----------	----------	EPA 9050A	EPA 9050A
Corrosivity	----------	----------	EPA 9040B	9045C
Ignitibility	----------	EPA 1010/EPA 1010A	EPA 1010 / 1010A	EPA 1010 / 1010A
Paint Filter Liquids Test	----------	----------	EPA 9095A	EPA 9095A

Parameter/Analyte	WY Storage Tank Program	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Synthetic Precipitation Leaching Procedure (SPLP)	----------	----------	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure	----------	----------	EPA 1311	EPA 1311
Organic Prep Methods				
Separatory Funnel Liquid-Liquid Extraction	----------	----------	EPA 3510C	----------
Continuous Liquid-Liquid Extraction	----------	----------	EPA 3520C	----------
Soxhlet Extraction	----------	----------	----------	EPA 3540C
Microwave Extraction	----------	----------	----------	EPA 3546
Ultrasonic Extraction	----------	----------	----------	EPA 3550B
Ultrasonic Extraction	----------	----------	----------	EPA 3550C
Waste Dilution	----------	----------	EPA 3580A	EPA 3580A
Solid Phase Extraction Volatiles Purge and trap Volatiles Purge and Trap for Soils	----------	----------	EPA 3535A EPA 5030B	EPA 5030B EPA 5035
Organic Cleanup Procedures				
Florisil Cleanup	----------	----------	EPA 3620B	EPA 3620B
Florisil Cleanup	----------	----------	EPA 3620C	EPA 3620C
Sulfur Cleanup	----------	----------	EPA 3660B	EPA 3660B
Sulfuric Acid/Permanganate Cleanup	----------	----------	EPA 3665A	EPA 3665A
Metals Digestion				
Acid Digestion Total Recoverable or Dissolved Metals	----------	----------	EPA 3005A	----------
Acid Digestion for Total Metals	----------	----------	EPA 3010A	----------
Acid Digestion for Total Metals	----------	----------	EPA 3020A	----------
Acid Digestion of Sediments, Sludges and Soils	----------	----------	----------	EPA 3050B

American Association for Laboratory Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality System Manual for Environmental Laboratories (QSM); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Presented this 5th day of November 2013.

President & CEO
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2015

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

State of California—Health and Human Services Agency
California Department of Public Health

RON CHAPMAN, MD, MPH
Director & State Health Officer

EDMUND G. BROWN JR.
Governor

May 23, 2014

Elaine Wild
Testamerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Dear Elaine Wild: Certificate No. 2886

This is to advise you that the laboratory named above has been granted an interim certificate pursuant to California Health and Safety Code (HSC), Division 101, Part 1, Chapter 4, Section 100850(d).

The Fields of Testing for which this laboratory has been granted interim certification is shown in the enclosed "Fields of Testing". The Interim certificate shall remain in effect until **March 31, 2015** or until a certificate pursuant to HSC 100825(a) is issued.

Your laboratory is required to participate in the appropriate performance evaluation studies and to perform acceptably in such studies as stated in HSC 100870 and Title 22 of the California Code of Regulations Section 64809. Continued compliance with the Environmental Laboratory Accreditation Program Branch statutes and regulations is required for maintaining the interim certification status.

Any changes in laboratory location or structural alterations, which may adversely affect the quality of analysis in the fields of testing for which the laboratory has been granted certification, require prior notification. Notification is also required for changes in ownership or laboratory director within 30 days after the change (HSC 100845(b) and (d)).

If you have any questions, please contact Jane Jensen at (510) 620-3174.

Sincerely,

Fred Chosbe for

David Mazzera, Ph.D., Assistant Division Chief
Division of Drinking Water and Environmental Management

Enclosure

CALIFORNIA DEPARTMENT OF PUBLIC HEALTH
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing

Testamerica St. Louis
13715 Rider Trail North
Earth City, MO 63045
Phone: (314) 298-8566

Certificate No.: 2886
Renew Date: 3/31/2015
INTERIM

Field of Testing: 106 - Radiochemistry of Drinking Water				
106.010	001	Gross Alpha	EPA 900.0	Interim
106.010	002	Gross Beta	EPA 900.0	Interim
106.030	001	Radioactive Cesium	EPA 901.1	Interim
106.030	003	Gamma Emitters	EPA 901.1	Interim
106.050	002	Radium-226	EPA 903.0	Interim
106.060	001	Radium-228	EPA 904.0	Interim
106.070	003	Strontium-90	EPA 905.0	Interim
106.080	001	Tritium	EPA 906.0	Interim
106.220	001	Strontium-89, 90	DOE Sr-02	Interim

Field of Testing: 108 - Inorganic Chemistry of Wastewater				
108.020	001	Conductivity	EPA 120.1	Interim
108.112	001	Boron	EPA 200.7	Interim
108.112	002	Calcium	EPA 200.7	Interim
108.112	004	Magnesium	EPA 200.7	Interim
108.112	005	Potassium	EPA 200.7	Interim
108.112	007	Sodium	EPA 200.7	Interim
108.113	001	Boron	EPA 200.8	Interim
108.113	002	Calcium	EPA 200.8	Interim
108.113	003	Magnesium	EPA 200.8	Interim
108.113	004	Potassium	EPA 200.8	Interim
108.113	006	Sodium	EPA 200.8	Interim
108.120	001	Bromide	EPA 300.0	Interim
108.120	002	Chloride	EPA 300.0	Interim
108.120	003	Fluoride	EPA 300.0	Interim
108.120	004	Nitrate	EPA 300.0	Interim
108.120	005	Nitrite	EPA 300.0	Interim
108.120	007	Phosphate, Ortho	EPA 300.0	Interim
108.120	008	Sulfate	EPA 300.0	Interim
108.183	001	Cyanide, Total	EPA 335.4	Interim
108.211	002	Kjeldahl Nitrogen, Total (as N)	EPA 351.2	Interim
108.323	001	Chemical Oxygen Demand	EPA 410.4	Interim
108.362	001	Phenols, Total	EPA 420.4	Interim
108.381	001	Oil and Grease	EPA 1664A	Interim

As of 04/01/2014 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

108.410	001	Alkalinity	SM2320B	Interim
108.440	001	Residue, Total	SM2540B	Interim
108.441	001	Residue, Filterable TDS	SM2540C	Interim
108.442	001	Residue, Non-filterable TSS	SM2540D	Interim
108.490	001	Hydrogen Ion (pH)	SM4500-H+ B	Interim

Field of Testing: 109 - Toxic Chemical Elements of Wastewater

109.010	001	Aluminum	EPA 200.7	Interim
109.010	002	Antimony	EPA 200.7	Interim
109.010	003	Arsenic	EPA 200.7	Interim
109.010	004	Barium	EPA 200.7	Interim
109.010	005	Beryllium	EPA 200.7	Interim
109.010	006	Boron	EPA 200.7	Interim
109.010	007	Cadmium	EPA 200.7	Interim
109.010	009	Chromium	EPA 200.7	Interim
109.010	010	Cobalt	EPA 200.7	Interim
109.010	011	Copper	EPA 200.7	Interim
109.010	012	Iron	EPA 200.7	Interim
109.010	013	Lead	EPA 200.7	Interim
109.010	015	Manganese	EPA 200.7	Interim
109.010	016	Molybdenum	EPA 200.7	Interim
109.010	017	Nickel	EPA 200.7	Interim
109.010	019	Selenium	EPA 200.7	Interim
109.010	021	Silver	EPA 200.7	Interim
109.010	023	Thallium	EPA 200.7	Interim
109.010	024	Tin	EPA 200.7	Interim
109.010	025	Titanium	EPA 200.7	Interim
109.010	026	Vanadium	EPA 200.7	Interim
109.010	027	Zinc	EPA 200.7	Interim
109.020	001	Aluminum	EPA 200.8	Interim
109.020	002	Antimony	EPA 200.8	Interim
109.020	003	Arsenic	EPA 200.8	Interim
109.020	004	Barium	EPA 200.8	Interim
109.020	005	Beryllium	EPA 200.8	Interim
109.020	006	Cadmium	EPA 200.8	Interim
109.020	007	Chromium	EPA 200.8	Interim
109.020	008	Cobalt	EPA 200.8	Interim
109.020	009	Copper	EPA 200.8	Interim
109.020	010	Lead	EPA 200.8	Interim
109.020	011	Manganese	EPA 200.8	Interim
109.020	012	Molybdenum	EPA 200.8	Interim

109.020	013	Nickel	EPA 200.8	Interim
109.020	014	Selenium	EPA 200.8	Interim
109.020	015	Silver	EPA 200.8	Interim
109.020	016	Thallium	EPA 200.8	Interim
109.020	017	Vanadium	EPA 200.8	Interim
109.020	018	Zinc	EPA 200.8	Interim
109.020	021	Iron	EPA 200.8	Interim
109.020	022	Tin	EPA 200.8	Interim
109.020	023	Titanium	EPA 200.8	Interim
109.190	001	Mercury	EPA 245.1	Interim

Field of Testing: 110 - Volatile Organic Chemistry of Wastewater

110.040	000	Purgeable Organic Compounds	EPA 624	Interim
110.040	029	Trichloroethene	EPA 624	Interim

Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater

111.100	000	Base/Neutral & Acid Organics	EPA 625	Interim
111.103	000	Nitrosamines	EPA 625	Interim
111.170	000	Organochlorine Pesticides	EPA 608	Interim

Field of Testing: 112 - Radiochemistry of Wastewater

112.010	001	Gross Alpha	EPA 900.0	Interim
112.010	002	Gross Beta	EPA 900.0	Interim
112.020	001	Total Alpha Radium	EPA 903.0	Interim
112.140	002	Gamma	EPA 901.1	Interim
112.160	001	Radium-228	EPA 904.0	Interim
112.170	001	Strontium	EPA 905.0	Interim
112.180	001	Tritium	EPA 906.0	Interim
112.510	001	Strontium	DOE Sr-02	Interim

Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste

114.010	001	Antimony	EPA 6010B	Interim
114.010	002	Arsenic	EPA 6010B	Interim
114.010	003	Barium	EPA 6010B	Interim
114.010	004	Beryllium	EPA 6010B	Interim
114.010	005	Cadmium	EPA 6010B	Interim
114.010	006	Chromium	EPA 6010B	Interim
114.010	007	Cobalt	EPA 6010B	Interim
114.010	008	Copper	EPA 6010B	Interim
114.010	009	Lead	EPA 6010B	Interim
114.010	010	Molybdenum	EPA 6010B	Interim
114.010	011	Nickel	EPA 6010B	Interim
114.010	012	Selenium	EPA 6010B	Interim
114.010	013	Silver	EPA 6010B	Interim

114.010	014	Thallium	EPA 6010B	Interim
114.010	015	Vanadium	EPA 6010B	Interim
114.010	016	Zinc	EPA 6010B	Interim
114.020	001	Antimony	EPA 6020	Interim
114.020	002	Arsenic	EPA 6020	Interim
114.020	003	Barium	EPA 6020	Interim
114.020	004	Beryllium	EPA 6020	Interim
114.020	005	Cadmium	EPA 6020	Interim
114.020	006	Chromium	EPA 6020	Interim
114.020	007	Cobalt	EPA 6020	Interim
114.020	008	Copper	EPA 6020	Interim
114.020	009	Lead	EPA 6020	Interim
114.020	010	Molybdenum	EPA 6020	Interim
114.020	011	Nickel	EPA 6020	Interim
114.020	012	Selenium	EPA 6020	Interim
114.020	013	Silver	EPA 6020	Interim
114.020	014	Thallium	EPA 6020	Interim
114.020	015	Vanadium	EPA 6020	Interim
114.020	016	Zinc	EPA 6020	Interim
114.103	001	Chromium (VI)	EPA 7196A	Interim
114.140	001	Mercury	EPA 7470A	Interim
114.141	001	Mercury	EPA 7471A	Interim
114.221	001	Cyanide, Total	EPA 9012A	Interim
114.240	001	Corrosivity - pH Determination	EPA 9040B	Interim
114.241	001	Corrosivity - pH Determination	EPA 9045C	Interim
114.250	001	Fluoride	EPA 9056	Interim
Field of Testing: 115 - Extraction Test of Hazardous Waste				
115.020	001	Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311	Interim
115.030	001	Waste Extraction Test (WET)	CCR Chapter11, Article 5, Appendix II	Interim
115.040	001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312	Interim
Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste				
116.030	001	Gasoline-range Organics	EPA 8015B	Interim
116.080	000	Volatile Organic Compounds	EPA 8260B	Interim
116.080	027	Dibromochloropropane	EPA 8260B	Interim
116.080	095	Xylenes, Total	EPA 8260B	Interim
Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste				
117.010	001	Diesel-range Total Petroleum Hydrocarbons	EPA 8015B	Interim
117.110	000	Extractable Organics	EPA 8270C	Interim
117.110	025	Carbazole	EPA 8270C	Interim
117.110	050	p-Dimethylaminoazobenzene	EPA 8270C	Interim

117.170	000	Nitroaromatics and Nitramines	EPA 8330	Interim
117.210	000	Organochlorine Pesticides	EPA 8081A	Interim
117.220	000	PCBs	EPA 8082	Interim
117.250	000	Chlorinated Herbicides	EPA 8151A	Interim

Field of Testing: 118 - Radiochemistry of Hazardous Waste

118.010	001	Gross Alpha	EPA 9310	Interim
118.010	002	Gross Beta	EPA 9310	Interim
118.020	001	Radium, Total	EPA 9315	Interim
118.030	001	Radium-228	EPA 9320	Interim
118.271	001	Strontium	DOE Sr-02	Interim

Field of Testing: 120 - Physical Properties of Hazardous Waste

120.010	001	Ignitability	EPA 1010	Interim
120.040	001	Reactive Cyanide	Section 7.3 SW-846	Interim
120.050	001	Reactive Sulfide	Section 7.3 SW-846	Interim
120.070	001	Corrosivity - pH Determination	EPA 9040B	Interim
120.080	001	Corrosivity - pH Determination	EPA 9045C	Interim

LABORATORY ACCREDITATION BUREAU

Certificate of Accreditation

ISO/IEC 17025:2005 **Certificate Number L2305**

TestAmerica Laboratories

St. Louis Facility
13715 Rider Trail North
Earth City Missouri 63045

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: January 10, 2016

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 31st of May 2013

*See the laboratory's Scope of Accreditation for details of accredited parameters
**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2305

Scope of Accreditation For TestAmerica Laboratories

St. Louis Facility
13715 Rider Trail North
Earth City, Missouri 63045
Marti Ward
314-298-8566

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.2) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to TestAmerica Laboratories, Inc. to perform the following tests:

Accreditation granted through: **January 10, 2016**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010C	Aluminum
ICP-AES	EPA 6010C	Antimony
ICP-AES	EPA 6010C	Arsenic
ICP-AES	EPA 6010C	Barium
ICP-AES	EPA 6010C	Beryllium
ICP-AES	EPA 6010C	Bismuth
ICP-AES	EPA 6010C	Boron
ICP-AES	EPA 6010C	Cadmium
ICP-AES	EPA 6010C	Calcium
ICP-AES	EPA 6010C	Chromium
ICP-AES	EPA 6010C	Cobalt
ICP-AES	EPA 6010C	Copper
ICP-AES	EPA 6010C	Iron
ICP-AES	EPA 6010C	Lead
ICP-AES	EPA 6010C	Lithium
ICP-AES	EPA 6010C	Magnesium

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010C	Manganese
ICP-AES	EPA 6010C	Molybdenum
ICP-AES	EPA 6010C	Nickel
ICP-AES	EPA 6010C	Phosphorus
ICP-AES	EPA 6010C	Potassium
ICP-AES	EPA 6010C	Selenium
ICP-AES	EPA 6010C	Silicon
ICP-AES	EPA 6010C	Silver
ICP-AES	EPA 6010C	Sodium
ICP-AES	EPA 6010C	Strontium
ICP-AES	EPA 6010C	Sulfur
ICP-AES	EPA 6010C	Thallium
ICP-AES	EPA 6010C	Thorium
ICP-AES	EPA 6010C	Tin
ICP-AES	EPA 6010C	Titanium
ICP-AES	EPA 6010C	Uranium
ICP-AES	EPA 6010C	Vanadium
ICP-AES	EPA 6010C	Zinc
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Benzyl chloride
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	n-Butanol
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Carbon disulfide

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chlorobromomethane
GC/MS	EPA 8260C	2-Chloro-1,3-butadiene
GC/MS	EPA 8260C	Chlorodibromomethane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Allyl chloride
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Cyclohexanone
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260C	1,2-Dibromoethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,2-Dichloroethene (total)
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	1,1-Dichloropropene

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	1,2-Dichloro-1,1,2,2-tetrafluoroethane
GC/MS	EPA 8260C	Dimethyl disulfide
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	Ethyl acetate
GC/MS	EPA 8260C	Ethylbenzene
GC/MS	EPA 8260C	Ethyl ether
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Freon 113
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	n-Hexane
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	p-Isopropyltoluene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methyl butyl ketone
GC/MS	EPA 8260C	Methylcyclohexane
GC/MS	EPA 8260C	Dichloromethane
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	4-Methyl-2-pentanone
GC/MS	EPA 8260C	MTBE
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	2-Nitropropane
GC/MS	EPA 8260C	Nonanal
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	Tetrachloroethene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260C	Trichlorotrifluoroethane
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	m-Xylene & p-Xylene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Xylenes (total)
GC/MS	EPA 8260C SIM	1,4-Dioxane
GC/MS	EPA 624	Acetone
GC/MS	EPA 624	Acetonitrile
GC/MS	EPA 624	Acrolein
GC/MS	EPA 624	Acrylonitrile
GC/MS	EPA 624	Benzene
GC/MS	EPA 624	Benzyl chloride
GC/MS	EPA 624	Bromobenzene
GC/MS	EPA 624	Bromochloromethane
GC/MS	EPA 624	Bromodichloromethane
GC/MS	EPA 624	Bromoform
GC/MS	EPA 624	Bromomethane
GC/MS	EPA 624	n-Butanol
GC/MS	EPA 624	2-Butanone
GC/MS	EPA 624	n-Butylbenzene
GC/MS	EPA 624	sec-Butylbenzene

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624	tert-Butylbenzene
GC/MS	EPA 624	Carbon disulfide
GC/MS	EPA 624	Carbon tetrachloride
GC/MS	EPA 624	Chlorobenzene
GC/MS	EPA 624	Chlorobromomethane
GC/MS	EPA 624	2-Chloro-1,3-butadiene
GC/MS	EPA 624	Chlorodibromomethane
GC/MS	EPA 624	Dibromochloromethane
GC/MS	EPA 624	Chloroethane
GC/MS	EPA 624	2-Chloroethyl vinyl ether
GC/MS	EPA 624	Chloroform
GC/MS	EPA 624	Chloromethane
GC/MS	EPA 624	Allyl chloride
GC/MS	EPA 624	2-Chlorotoluene
GC/MS	EPA 624	4-Chlorotoluene
GC/MS	EPA 624	Cyclohexane
GC/MS	EPA 624	Cyclohexanone
GC/MS	EPA 624	1,2-Dibromo-3-chloropropane
GC/MS	EPA 624	1,2-Dibromoethane
GC/MS	EPA 624	Dibromomethane
GC/MS	EPA 624	1,2-Dichlorobenzene
GC/MS	EPA 624	1,3-Dichlorobenzene
GC/MS	EPA 624	1,4-Dichlorobenzene
GC/MS	EPA 624	trans-1,4-Dichloro-2-butene
GC/MS	EPA 624	Dichlorodifluoromethane
GC/MS	EPA 624	1,1-Dichloroethane
GC/MS	EPA 624	1,2-Dichloroethane
GC/MS	EPA 624	cis-1,2-Dichloroethene
GC/MS	EPA 624	trans-1,2-Dichloroethene
GC/MS	EPA 624	1,1-Dichloroethene
GC/MS	EPA 624	1,2-Dichloroethene (total)
GC/MS	EPA 624	1,2-Dichloropropane
GC/MS	EPA 624	1,3-Dichloropropane
GC/MS	EPA 624	2,2-Dichloropropane
GC/MS	EPA 624	cis-1,3-Dichloropropene

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624	trans-1,3-Dichloropropene
GC/MS	EPA 624	1,1-Dichloropropene
GC/MS	EPA 624	1,2-Dichloro-1,1,2,2-tetrafluoroethane
GC/MS	EPA 624	Dimethyl disulfide
GC/MS	EPA 624	1,4-Dioxane
GC/MS	EPA 624	Ethyl acetate
GC/MS	EPA 624	Ethylbenzene
GC/MS	EPA 624	Ethyl ether
GC/MS	EPA 624	Diethyl ether
GC/MS	EPA 624	Ethyl methacrylate
GC/MS	EPA 624	Freon 113
GC/MS	EPA 624	Hexachlorobutadiene
GC/MS	EPA 624	n-Hexane
GC/MS	EPA 624	2-Hexanone
GC/MS	EPA 624	Iodomethane
GC/MS	EPA 624	Isobutanol
GC/MS	EPA 624	Isopropylbenzene
GC/MS	EPA 624	p-Isopropyltoluene
GC/MS	EPA 624	Methacrylonitrile
GC/MS	EPA 624	Methyl acetate
GC/MS	EPA 624	Methyl butyl ketone
GC/MS	EPA 624	Methylcyclohexane
GC/MS	EPA 624	Dichloromethane
GC/MS	EPA 624	Methylene chloride
GC/MS	EPA 624	Methyl methacrylate
GC/MS	EPA 624	4-Methyl-2-pentanone
GC/MS	EPA 624	MTBE
GC/MS	EPA 624	Naphthalene
GC/MS	EPA 624	2-Nitropropane
GC/MS	EPA 624	Nonanal
GC/MS	EPA 624	Pentachloroethane
GC/MS	EPA 624	Propionitrile
GC/MS	EPA 624	n-Propylbenzene
GC/MS	EPA 624	Styrene
GC/MS	EPA 624	1,1,1,2-Tetrachloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624	1,1,2,2-Tetrachloroethane
GC/MS	EPA 624	Tetrachloroethene
GC/MS	EPA 624	Tetrahydrofuran
GC/MS	EPA 624	Toluene
GC/MS	EPA 624	1,3,5-Trichlorobenzene
GC/MS	EPA 624	1,2,3-Trichlorobenzene
GC/MS	EPA 624	1,2,4-Trichlorobenzene
GC/MS	EPA 624	1,1,1-Trichloroethane
GC/MS	EPA 624	1,1,2-Trichloroethane
GC/MS	EPA 624	Trichloroethene
GC/MS	EPA 624	Trichlorofluoromethane
GC/MS	EPA 624	1,2,3-Trichloropropane
GC/MS	EPA 624	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 624	Trichlorotrifluoroethane
GC/MS	EPA 624	1,2,4-Trimethylbenzene
GC/MS	EPA 624	1,3,5-Trimethylbenzene
GC/MS	EPA 624	Vinyl acetate
GC/MS	EPA 624	Vinyl chloride
GC/MS	EPA 624	m-Xylene & p-Xylene
GC/MS	EPA 624	o-Xylene
GC/MS	EPA 624	Xylenes (total)
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite (total)
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Azobenzene
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo(a)anthracene
GC/MS	EPA 8270D	Benzo(b)fluoranthene

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270D	Benzoic acid
GC/MS	EPA 8270D	Benzo(ghi)perylene
GC/MS	EPA 8270D	Benzo(a)pyrene
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	1,1'-Biphenyl
GC/MS	EPA 8270D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270D	bis(2-Chloroisopropyl) ether
GC/MS	EPA 8270D	bis(2-Ethylhexyl) phthalate
GC/MS	EPA 8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270D	n-Butylbenzenesulfonamide
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	Chlorobenzilate
GC/MS	EPA 8270D	p-Chlorobenzilate
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols (total)
GC/MS	EPA 8270D	Cyclohexanol
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dichlorophenol

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Diethyl phthalate
GC/MS	EPA 8270D	O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	Dimethylformamide
GC/MS	EPA 8270D	alpha,alpha-Dimethylphenethylamine
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dinitrobenzene
GC/MS	EPA 8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	Dinoseb
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methacrylate
GC/MS	EPA 8270D	Ethyl methanesulfonate
GC/MS	EPA 8270D	Famphur
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorobutadiene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloro-1,3-cyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D	Isodrin

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	Isosafrole
GC/MS	EPA 8270D	Kepone
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	2-Methylbenzenamine
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	4,4'-Methylenebis(2-chloroaniline)
GC/MS	EPA 8270D	Methyl methacrylate
GC/MS	EPA 8270D	Methyl methanesulfonate
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	Methyl parathion
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	3-Methylphenol & 4-Methylphenol
GC/MS	EPA 8270D	2-Methylphenol, 3-methylphenol and 4-methylphenol
GC/MS	EPA 8270D	Methylphenols (total)
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Naphthylamine
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	N-Nitrosodi-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine
GC/MS	EPA 8270D	5-Nitro-o-toluidine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	2,2'-oxybis(1-Chloropropane)
GC/MS	EPA 8270D	Parathion
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachloroethane
GC/MS	EPA 8270D	Pentachloronitrobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	2-Picoline
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	Tetraethyldithiopyrophosphate (Sulfotepp)
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270D	o-Toluidine
GC/MS	EPA 8270D	Tributyl phosphate
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	Tris(2-chloroethyl)phosphate
GC/MS	EPA 8270D	1-Methyl naphthalene
GC/MS	EPA 625	Acenaphthene
GC/MS	EPA 625	Acenaphthylene
GC/MS	EPA 625	Acetophenone
GC/MS	EPA 625	2-Acetylaminofluorene
GC/MS	EPA 625	4-Aminobiphenyl
GC/MS	EPA 625	Aniline

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625	Anthracene
GC/MS	EPA 625	Aramite (total)
GC/MS	EPA 625	Atrazine
GC/MS	EPA 625	Azobenzene
GC/MS	EPA 625	Benzaldehyde
GC/MS	EPA 625	Benzidine
GC/MS	EPA 625	Benzo(a)anthracene
GC/MS	EPA 625	Benzo(b)fluoranthene
GC/MS	EPA 625	Benzo(k)fluoranthene
GC/MS	EPA 625	Benzoic acid
GC/MS	EPA 625	Benzo(ghi)perylene
GC/MS	EPA 625	Benzo(a)pyrene
GC/MS	EPA 625	Benzyl alcohol
GC/MS	EPA 625	1,1'-Biphenyl
GC/MS	EPA 625	bis(2-Chloroethoxy)methane
GC/MS	EPA 625	bis(2-Chloroethyl) ether
GC/MS	EPA 625	bis(2-Chloroisopropyl) ether
GC/MS	EPA 625	bis(2-Ethylhexyl) phthalate
GC/MS	EPA 625	4-Bromophenyl phenyl ether
GC/MS	EPA 625	n-Butylbenzenesulfonamide
GC/MS	EPA 625	Butyl benzyl phthalate
GC/MS	EPA 625	Caprolactam
GC/MS	EPA 625	Carbazole
GC/MS	EPA 625	4-Chloroaniline
GC/MS	EPA 625	Chlorobenzilate
GC/MS	EPA 625	p-Chlorobenzilate
GC/MS	EPA 625	4-Chloro-3-methylphenol
GC/MS	EPA 625	2-Chloronaphthalene
GC/MS	EPA 625	2-Chlorophenol
GC/MS	EPA 625	4-Chlorophenyl phenyl ether
GC/MS	EPA 625	Chrysene
GC/MS	EPA 625	Cresols (total)
GC/MS	EPA 625	Cyclohexanol
GC/MS	EPA 625	Diallate
GC/MS	EPA 625	Dibenz(a,h)anthracene
GC/MS	EPA 625	Dibenzo(a,h)anthracene
GC/MS	EPA 625	Dibenzofuran
GC/MS	EPA 625	Di-n-butyl phthalate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625	1,2-Dichlorobenzene
GC/MS	EPA 625	1,3-Dichlorobenzene
GC/MS	EPA 625	1,4-Dichlorobenzene
GC/MS	EPA 625	3,3'-Dichlorobenzidine
GC/MS	EPA 625	2,4-Dichlorophenol
GC/MS	EPA 625	2,6-Dichlorophenol
GC/MS	EPA 625	Diethyl phthalate
GC/MS	EPA 625	O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate
GC/MS	EPA 625	Dimethoate
GC/MS	EPA 625	p-Dimethylaminoazobenzene
GC/MS	EPA 625	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 625	3,3'-Dimethylbenzidine
GC/MS	EPA 625	Dimethylformamide
GC/MS	EPA 625	alpha,alpha-Dimethylphenethylamine
GC/MS	EPA 625	2,4-Dimethylphenol
GC/MS	EPA 625	Dimethyl phthalate
GC/MS	EPA 625	1,3-Dinitrobenzene
GC/MS	EPA 625	1,4-Dinitrobenzene
GC/MS	EPA 625	4,6-Dinitro-2-methylphenol
GC/MS	EPA 625	2,4-Dinitrophenol
GC/MS	EPA 625	2,4-Dinitrotoluene
GC/MS	EPA 625	2,6-Dinitrotoluene
GC/MS	EPA 625	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 625	Dinoseb
GC/MS	EPA 625	Di-n-octyl phthalate
GC/MS	EPA 625	1,4-Dioxane
GC/MS	EPA 625	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 625	Disulfoton
GC/MS	EPA 625	Ethyl methacrylate
GC/MS	EPA 625	Ethyl methanesulfonate
GC/MS	EPA 625	Famphur
GC/MS	EPA 625	Fluoranthene
GC/MS	EPA 625	Fluorene
GC/MS	EPA 625	Hexachlorobenzene
GC/MS	EPA 625	Hexachlorobutadiene
GC/MS	EPA 625	Hexachlorocyclopentadiene
GC/MS	EPA 625	Hexachloro-1,3-cyclopentadiene
GC/MS	EPA 625	Hexachloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625	Hexachlorophene
GC/MS	EPA 625	Hexachloropropene
GC/MS	EPA 625	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 625	Isodrin
GC/MS	EPA 625	Isophorone
GC/MS	EPA 625	Isosafrole
GC/MS	EPA 625	Kepone
GC/MS	EPA 625	Methapyrilene
GC/MS	EPA 625	2-Methylbenzenamine
GC/MS	EPA 625	3-Methylcholanthrene
GC/MS	EPA 625	4,4'-Methylenebis(2-chloroaniline)
GC/MS	EPA 625	Methyl methacrylate
GC/MS	EPA 625	Methyl methanesulfonate
GC/MS	EPA 625	2-Methylnaphthalene
GC/MS	EPA 625	Methyl parathion
GC/MS	EPA 625	2-Methylphenol
GC/MS	EPA 625	3-Methylphenol & 4-Methylphenol
GC/MS	EPA 625	2-Methylphenol, 3-methylphenol and 4-methylphenol
GC/MS	EPA 625	Methylphenols (total)
GC/MS	EPA 625	Naphthalene
GC/MS	EPA 625	1,4-Naphthoquinone
GC/MS	EPA 625	1-Naphthylamine
GC/MS	EPA 625	2-Naphthylamine
GC/MS	EPA 625	2-Nitroaniline
GC/MS	EPA 625	3-Nitroaniline
GC/MS	EPA 625	4-Nitroaniline
GC/MS	EPA 625	Nitrobenzene
GC/MS	EPA 625	2-Nitrophenol
GC/MS	EPA 625	4-Nitrophenol
GC/MS	EPA 625	4-Nitroquinoline-1-oxide
GC/MS	EPA 625	N-Nitrosodi-n-butylamine
GC/MS	EPA 625	N-Nitrosodiethylamine
GC/MS	EPA 625	N-Nitrosodimethylamine
GC/MS	EPA 625	N-Nitrosodiphenylamine
GC/MS	EPA 625	N-Nitrosodi-n-propylamine
GC/MS	EPA 625	N-Nitrosomethylethylamine
GC/MS	EPA 625	N-Nitrosomorpholine
GC/MS	EPA 625	N-Nitrosopiperidine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625	N-Nitrosopyrrolidine
GC/MS	EPA 625	5-Nitro-o-toluidine
GC/MS	EPA 625	2,2'-oxybis(1-Chloropropane)
GC/MS	EPA 625	Parathion
GC/MS	EPA 625	Pentachlorobenzene
GC/MS	EPA 625	Pentachloroethane
GC/MS	EPA 625	Pentachloronitrobenzene
GC/MS	EPA 625	Pentachlorophenol
GC/MS	EPA 625	Phenacetin
GC/MS	EPA 625	Phenanthrene
GC/MS	EPA 625	Phenol
GC/MS	EPA 625	p-Phenylene diamine
GC/MS	EPA 625	Phorate
GC/MS	EPA 625	2-Picoline
GC/MS	EPA 625	Pronamide
GC/MS	EPA 625	Pyrene
GC/MS	EPA 625	Pyridine
GC/MS	EPA 625	Safrole
GC/MS	EPA 625	Sulfotepp
GC/MS	EPA 625	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 625	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 625	Tetraethyldithiopyrophosphate (Sulfotepp)
GC/MS	EPA 625	Thionazin
GC/MS	EPA 625	o-Toluidine
GC/MS	EPA 625	Tributyl phosphate
GC/MS	EPA 625	1,2,4-Trichlorobenzene
GC/MS	EPA 625	2,4,5-Trichlorophenol
GC/MS	EPA 625	2,4,6-Trichlorophenol
GC/MS	EPA 625	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 625	1,3,5-Trinitrobenzene
GC/MS	EPA 625	Tris(2-chloroethyl)phosphate
GC/MS	EPA 625	1-Methyl naphthalene
GC-ECD	EPA 8081B	Aldrin
GC-ECD	EPA 8081B	alpha-BHC
GC-ECD	EPA 8081B	beta-BHC
GC-ECD	EPA 8081B	delta-BHC
GC-ECD	EPA 8081B	gamma-BHC (Lindane)
GC-ECD	EPA 8081B	alpha-Chlordane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081B	gamma-Chlordane
GC-ECD	EPA 8081B	Chlordane (technical)
GC-ECD	EPA 8081B	4,4'-DDD
GC-ECD	EPA 8081B	2,4'-DDD
GC-ECD	EPA 8081B	4,4'-DDE
GC-ECD	EPA 8081B	2,4'-DDE
GC-ECD	EPA 8081B	4,4'-DDT
GC-ECD	EPA 8081B	2,4'-DDT
GC-ECD	EPA 8081B	Dieldrin
GC-ECD	EPA 8081B	Endosulfan I
GC-ECD	EPA 8081B	Endosulfan II
GC-ECD	EPA 8081B	Endosulfan sulfate
GC-ECD	EPA 8081B	Endrin
GC-ECD	EPA 8081B	Endrin aldehyde
GC-ECD	EPA 8081B	Endrin ketone
GC-ECD	EPA 8081B	Heptachlor
GC-ECD	EPA 8081B	Heptachlor epoxide
GC-ECD	EPA 8081B	Methoxychlor
GC-ECD	EPA 8081B	Toxaphene
GC-ECD	EPA 608	Aldrin
GC-ECD	EPA 608	alpha-BHC
GC-ECD	EPA 608	beta-BHC
GC-ECD	EPA 608	delta-BHC
GC-ECD	EPA 608	gamma-BHC (Lindane)
GC-ECD	EPA 608	alpha-Chlordane
GC-ECD	EPA 608	gamma-Chlordane
GC-ECD	EPA 608	Chlordane (technical)
GC-ECD	EPA 608	4,4'-DDD
GC-ECD	EPA 608	2,4'-DDD
GC-ECD	EPA 608	4,4'-DDE
GC-ECD	EPA 608	2,4'-DDE
GC-ECD	EPA 608	4,4'-DDT
GC-ECD	EPA 608	2,4'-DDT
GC-ECD	EPA 608	Dieldrin
GC-ECD	EPA 608	Endosulfan I
GC-ECD	EPA 608	Endosulfan II

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 608	Endosulfan sulfate
GC-ECD	EPA 608	Endrin
GC-ECD	EPA 608	Endrin aldehyde
GC-ECD	EPA 608	Endrin ketone
GC-ECD	EPA 608	Heptachlor
GC-ECD	EPA 608	Heptachlor epoxide
GC-ECD	EPA 608	Methoxychlor
GC-ECD	EPA 608	Toxaphene
GC-ECD	EPA 608	Aroclor 1016
GC-ECD	EPA 608	Aroclor 1221
GC-ECD	EPA 608	Aroclor 1232
GC-ECD	EPA 608	Aroclor 1242
GC-ECD	EPA 608	Aroclor 1248
GC-ECD	EPA 608	Aroclor 1254
GC-ECD	EPA 608	Aroclor 1260
GC-ECD	EPA 608	Aroclor 1262
GC-ECD	EPA 608	Aroclor 1268
GC-ECD	EPA 8082A	Aroclor 1016
GC-ECD	EPA 8082A	Aroclor 1221
GC-ECD	EPA 8082A	Aroclor 1232
GC-ECD	EPA 8082A	Aroclor 1242
GC-ECD	EPA 8082A	Aroclor 1248
GC-ECD	EPA 8082A	Aroclor 1254
GC-ECD	EPA 8082A	Aroclor 1260
GC-ECD	EPA 8082A	Aroclor 1262
GC-ECD	EPA 8082A	Aroclor 1268
GC-ECD	EPA 8151A	2,4-D
GC-ECD	EPA 8151A	Dalapon
GC-ECD	EPA 8151A	2,4-DB
GC-ECD	EPA 8151A	Dicamba
GC-ECD	EPA 8151A	Dichlorprop
GC-ECD	EPA 8151A	Dinoseb
GC-ECD	EPA 8151A	MCPA
GC-ECD	EPA 8151A	MCPP
GC-ECD	EPA 8151A	4-Nitrophenol
GC-ECD	EPA 8151A	Pentachlorophenol

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8151A	2,4,5-TP (Silvex)
GC-ECD	EPA 8151A	2,4,5-T
LC/MS/MS	EPA 8321A	2,4-D
LC/MS/MS	EPA 8321A	Dalapon
LC/MS/MS	EPA 8321A	2,4-DB
LC/MS/MS	EPA 8321A	Dicamba
LC/MS/MS	EPA 8321A	Dichlorprop
LC/MS/MS	EPA 8321A	Dinoseb
LC/MS/MS	EPA 8321A	MCPA
LC/MS/MS	EPA 8321A	MCPP
LC/MS/MS	EPA 8321A	2,4,5-TP (Silvex)
LC/MS/MS	EPA 8321A	2,4,5-T
GC-FID	RSK-175	Methane
GC-FID	RSK-175	Ethane
GC-FID	RSK-175	Ethene
GC-FID	RSK-175	Acetylene
GC-FID	EPA 8015B	Ethanol
GC-FID	EPA 8015B	Methanol
GC-FID	EPA 8015B	Ethylene glycol
GC-FID	EPA 8015B	Propylene glycol
GC-FID	EPA 8015B	Diesel Range Organics
GC-FID	EPA 8015B	Motor Oil Range Organics
GC-FID	EPA 8015B	TPH (as Diesel)
GC-FID	EPA 8015B	Gasoline Range Organics
LC/MS/MS	EPA 8321A	2-Amino-4,6-dinitrotoluene
LC/MS/MS	EPA 8321A	4-Amino-2,6-dinitrotoluene
LC/MS/MS	EPA 8321A	3,5-Dinitroaniline
LC/MS/MS	EPA 8321A	1,3-Dinitrobenzene
LC/MS/MS	EPA 8321A	2,4-Dinitrotoluene
LC/MS/MS	EPA 8321A	2,6-Dinitrotoluene
LC/MS/MS	EPA 8321A	DNX
LC/MS/MS	EPA 8321A	HMX
LC/MS/MS	EPA 8321A	HNAB
LC/MS/MS	EPA 8321A	HNS
LC/MS/MS	EPA 8321A	MNX
LC/MS/MS	EPA 8321A	Nitrobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 8321A	Nitroglycerin
LC/MS/MS	EPA 8321A	4-Nitrotoluene
LC/MS/MS	EPA 8321A	3-Nitrotoluene
LC/MS/MS	EPA 8321A	2-Nitrotoluene
LC/MS/MS	EPA 8321A	PETN
LC/MS/MS	EPA 8321A	RDX
LC/MS/MS	EPA 8321A	TATB
LC/MS/MS	EPA 8321A	Tetryl
LC/MS/MS	EPA 8321A	TNX
LC/MS/MS	EPA 8321A	1,3,5-Trinitrobenzene
LC/MS/MS	EPA 8321A	2,4,6-Trinitrotoluene
LC/MS/MS	EPA 8321A	Tris (o-cresyl) Phosphate
LC/MS/MS	EPA 8321A	2,4-diamino-6-nitrotoluene
LC/MS/MS	EPA 8321A	2,6-diamino-4-nitrotoluene
HPLC	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC	EPA 8330B	1,3-Dinitrobenzene
HPLC	EPA 8330B	2,4-Dinitrotoluene
HPLC	EPA 8330B	2,6-Dinitrotoluene
HPLC	EPA 8330B	HMX
HPLC	EPA 8330B	HNAB
HPLC	EPA 8330B	HNS
HPLC	EPA 8330B	Nitrobenzene
HPLC	EPA 8330B	Nitroglycerin
HPLC	EPA 8330B	2-Nitrotoluene
HPLC	EPA 8330B	3-Nitrotoluene
HPLC	EPA 8330B	4-Nitrotoluene
HPLC	EPA 8330B	PETN
HPLC	EPA 8330B	RDX
HPLC	EPA 8330B	TATB
HPLC	EPA 8330B	Tetryl
HPLC	EPA 8330B	MNX
HPLC	EPA 8330B	DNX
HPLC	EPA 8330B	TNX
HPLC	EPA 8330B	1,3,5-Trinitrobenzene
HPLC	EPA 8330B	2,4,6-Trinitrotoluene

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D SIM	Acenaphthene
GC/MS	EPA 8270D SIM	Acenaphthylene
GC/MS	EPA 8270D SIM	Anthracene
GC/MS	EPA 8270D SIM	Benzo(a)anthracene
GC/MS	EPA 8270D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270D SIM	Benzo(ghi)perylene
GC/MS	EPA 8270D SIM	Benzo(a)pyrene
GC/MS	EPA 8270D SIM	Chrysene
GC/MS	EPA 8270D SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D SIM	Fluoranthene
GC/MS	EPA 8270D SIM	Fluorene
GC/MS	EPA 8270D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D SIM	Naphthalene
GC/MS	EPA 8270D SIM	Phenanthrene
GC/MS	EPA 8270D SIM	Pyrene
LC/MS/MS	EPA 6850	Perchlorate
ICP-MS	EPA 6020A	Aluminum
ICP-MS	EPA 6020A	Antimony
ICP-MS	EPA 6020A	Arsenic
ICP-MS	EPA 6020A	Barium
ICP-MS	EPA 6020A	Beryllium
ICP-MS	EPA 6020A	Bismuth
ICP-MS	EPA 6020A	Boron
ICP-MS	EPA 6020A	Cadmium
ICP-MS	EPA 6020A	Calcium
ICP-MS	EPA 6020A	Cerium
ICP-MS	EPA 6020A	Cesium
ICP-MS	EPA 6020A	Chromium
ICP-MS	EPA 6020A	Cobalt
ICP-MS	EPA 6020A	Copper
ICP-MS	EPA 6020A	Hafnium
ICP-MS	EPA 6020A	Iron
ICP-MS	EPA 6020A	Lanthanum
ICP-MS	EPA 6020A	Lead
ICP-MS	EPA 6020A	Lithium

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020A	Magnesium
ICP-MS	EPA 6020A	Manganese
ICP-MS	EPA 6020A	Molybdenum
ICP-MS	EPA 6020A	Neodymium
ICP-MS	EPA 6020A	Nickel
ICP-MS	EPA 6020A	Niobium
ICP-MS	EPA 6020A	Palladium
ICP-MS	EPA 6020A	Phosphorus
ICP-MS	EPA 6020A	Platinum
ICP-MS	EPA 6020A	Potassium
ICP-MS	EPA 6020A	Praseodymium
ICP-MS	EPA 6020A	Rhodium
ICP-MS	EPA 6020A	Ruthenium
ICP-MS	EPA 6020A	Samarium
ICP-MS	EPA 6020A	Selenium
ICP-MS	EPA 6020A	Silicon
ICP-MS	EPA 6020A	Silver
ICP-MS	EPA 6020A	Sodium
ICP-MS	EPA 6020A	Strontium
ICP-MS	EPA 6020A	Sulfur
ICP-MS	EPA 6020A	Tantalum
ICP-MS	EPA 6020A	Tellurium
ICP-MS	EPA 6020A	Thallium
ICP-MS	EPA 6020A	Thorium
ICP-MS	EPA 6020A	Tin
ICP-MS	EPA 6020A	Titanium
ICP-MS	EPA 6020A	Tungsten
ICP-MS	EPA 6020A	Uranium
ICP-MS	EPA 6020A	Uranium 233
ICP-MS	EPA 6020A	Uranium 234
ICP-MS	EPA 6020A	Uranium 235
ICP-MS	EPA 6020A	Uranium 236
ICP-MS	EPA 6020A	Uranium 238
ICP-MS	EPA 6020A	Vanadium
ICP-MS	EPA 6020A	Yttrium
ICP-MS	EPA 6020A	Zinc

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020A	Zirconium
ICP-MS	EPA 200.8	Aluminum
ICP-MS	EPA 200.8	Antimony
ICP-MS	EPA 200.8	Arsenic
ICP-MS	EPA 200.8	Barium
ICP-MS	EPA 200.8	Beryllium
ICP-MS	EPA 200.8	Bismuth
ICP-MS	EPA 200.8	Boron
ICP-MS	EPA 200.8	Cadmium
ICP-MS	EPA 200.8	Calcium
ICP-MS	EPA 200.8	Cerium
ICP-MS	EPA 200.8	Cesium
ICP-MS	EPA 200.8	Chromium
ICP-MS	EPA 200.8	Cobalt
ICP-MS	EPA 200.8	Copper
ICP-MS	EPA 200.8	Hafnium
ICP-MS	EPA 200.8	Iron
ICP-MS	EPA 200.8	Lanthanum
ICP-MS	EPA 200.8	Lead
ICP-MS	EPA 200.8	Lithium
ICP-MS	EPA 200.8	Magnesium
ICP-MS	EPA 200.8	Manganese
ICP-MS	EPA 200.8	Molybdenum
ICP-MS	EPA 200.8	Neodymium
ICP-MS	EPA 200.8	Nickel
ICP-MS	EPA 200.8	Niobium
ICP-MS	EPA 200.8	Palladium
ICP-MS	EPA 200.8	Phosphorus
ICP-MS	EPA 200.8	Platinum
ICP-MS	EPA 200.8	Potassium
ICP-MS	EPA 200.8	Praseodymium
ICP-MS	EPA 200.8	Rhodium
ICP-MS	EPA 200.8	Ruthenium
ICP-MS	EPA 200.8	Samarium
ICP-MS	EPA 200.8	Selenium
ICP-MS	EPA 200.8	Silicon

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 200.8	Silver
ICP-MS	EPA 200.8	Sodium
ICP-MS	EPA 200.8	Strontium
ICP-MS	EPA 200.8	Sulfur
ICP-MS	EPA 200.8	Tantalum
ICP-MS	EPA 200.8	Tellurium
ICP-MS	EPA 200.8	Thallium
ICP-MS	EPA 200.8	Thorium
ICP-MS	EPA 200.8	Tin
ICP-MS	EPA 200.8	Titanium
ICP-MS	EPA 200.8	Tungsten
ICP-MS	EPA 200.8	Uranium
ICP-MS	EPA 200.8	Vanadium
ICP-MS	EPA 200.8	Yttrium
ICP-MS	EPA 200.8	Zinc
ICP-MS	EPA 200.8	Zirconium
ICP-AES	EPA 200.7	Aluminum
ICP-AES	EPA 200.7	Antimony
ICP-AES	EPA 200.7	Arsenic
ICP-AES	EPA 200.7	Barium
ICP-AES	EPA 200.7	Beryllium
ICP-AES	EPA 200.7	Bismuth
ICP-AES	EPA 200.7	Boron
ICP-AES	EPA 200.7	Cadmium
ICP-AES	EPA 200.7	Calcium
ICP-AES	EPA 200.7	Chromium
ICP-AES	EPA 200.7	Cobalt
ICP-AES	EPA 200.7	Copper
ICP-AES	EPA 200.7	Iron
ICP-AES	EPA 200.7	Lead
ICP-AES	EPA 200.7	Lithium
ICP-AES	EPA 200.7	Magnesium
ICP-AES	EPA 200.7	Manganese
ICP-AES	EPA 200.7	Molybdenum
ICP-AES	EPA 200.7	Nickel
ICP-AES	EPA 200.7	Phosphorus

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 200.7	Potassium
ICP-AES	EPA 200.7	Selenium
ICP-AES	EPA 200.7	Silicon
ICP-AES	EPA 200.7	Silver
ICP-AES	EPA 200.7	Sodium
ICP-AES	EPA 200.7	Strontium
ICP-AES	EPA 200.7	Sulfur
ICP-AES	EPA 200.7	Thallium
ICP-AES	EPA 200.7	Thorium
ICP-AES	EPA 200.7	Tin
ICP-AES	EPA 200.7	Titanium
ICP-AES	EPA 200.7	Uranium
ICP-AES	EPA 200.7	Vanadium
ICP-AES	EPA 200.7	Zinc
CVAA	EPA 7470A	Mercury
Colormetric	EPA 9010C EPA 9012B	Cyanide
Ion Chromatrography	EPA 300.0/9056A	Bromide
Ion Chromatrography	EPA 300.0/9056A	Chloride
Ion Chromatrography	EPA 300.0/9056A	Fluoride
Ion Chromatrography	EPA 300.0/9056A	Nitrate
Ion Chromatrography	EPA 300.0/9056A	Nitrite
Ion Chromatrography	EPA 300.0/9056A	Sulfate
Ion Chromatrography	EPA 300.0/9056A	Ortho-phosph
Ion Chromatrography	EPA 300.0/9056A	Iodide
Ion Chromatrography	EPA 314.0	Perchlorate
Gravimetric	SM 2540B SM 2540C SM 2540D	Solids
Probe	EPA 9040C EPA 9045D EPA 150.1	pH
Titration	SM 2320B EPA 310.1	Alkalinity
Titration	EPA 9030	Sulfide
Penske-Martin	EPA 1010A	Ignitability

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Non-Potable Water		
Technology	**Method**	**Analyte**
Colormetric	EPA 353.1	nitrate/Nitrite
Colormetric	EPA 350.1	Ammonia
Colormetric	EPA 351.2	TKN
TOC Analyzer	EPA 9060A	TOC
Tritrmetric	EPA 9020B	TOX
Colormetric	EPA 7196A	Hex Chromium
Gravimetric	EPA 1664A	Oil & Grease
Gravimetric	EPA 1664A	TPH
Probe	EPA 9050A	Conductivity
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 / DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium-137

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Calcium-45
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese-56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 83
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48

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Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Uranium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	Laboratory SOP ST-RC-0210	Polonium-210
Liquid Scintillation Counter	Eichrom Technologies FEW01	Iron-55
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129
Preparation	**Method**	**Type**
Organic Extraction & Sample Prep	EPA 3500C	Organic Extraction & Sample Prep
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Organic Cleanup	EPA 3600A	Cleanup for Organic extracts
Organic prep/analysis	EPA 8000C	Determinative Chromatographic Separations
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile

Non-Potable Water		
Preparation	**Method**	**Type**
Sep Funnel Liquid-Liquid Extraction	EPA 3510C	Sep Funnel Liquid-Liquid Extraction
Organic Cleanup	EPA 3600A	Cleanup for Organic extracts
Florisil Cleanup	EPA 3620C	Florisil Cleanup
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup
Acid Clean Up	EPA 3665A	Acid Clean Up for PCBs
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction
Solid Phase Extraction	EPA 3535A	Solid Phase Extraction

Drinking Water		
Technology	**Method**	**Analyte**
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 / DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	EPA 906.0	Tritium
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)

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Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium-137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Calcium-45
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137

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Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese-56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 83
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010C	Aluminum
ICP-AES	EPA 6010C	Antimony
ICP-AES	EPA 6010C	Arsenic
ICP-AES	EPA 6010C	Barium
ICP-AES	EPA 6010C	Beryllium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010C	Bismuth
ICP-AES	EPA 6010C	Boron
ICP-AES	EPA 6010C	Cadmium
ICP-AES	EPA 6010C	Calcium
ICP-AES	EPA 6010C	Chromium
ICP-AES	EPA 6010C	Cobalt
ICP-AES	EPA 6010C	Copper
ICP-AES	EPA 6010C	Iron
ICP-AES	EPA 6010C	Lead
ICP-AES	EPA 6010C	Lithium
ICP-AES	EPA 6010C	Magnesium
ICP-AES	EPA 6010C	Manganese
ICP-AES	EPA 6010C	Molybdenum
ICP-AES	EPA 6010C	Nickel
ICP-AES	EPA 6010C	Phosphorus
ICP-AES	EPA 6010C	Potassium
ICP-AES	EPA 6010C	Selenium
ICP-AES	EPA 6010C	Silicon
ICP-AES	EPA 6010C	Silver
ICP-AES	EPA 6010C	Sodium
ICP-AES	EPA 6010C	Strontium
ICP-AES	EPA 6010C	Sulfur
ICP-AES	EPA 6010C	Thallium
ICP-AES	EPA 6010C	Thorium
ICP-AES	EPA 6010C	Tin
ICP-AES	EPA 6010C	Titanium
ICP-AES	EPA 6010C	Uranium
ICP-AES	EPA 6010C	Vanadium
ICP-AES	EPA 6010C	Zinc
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Benzyl chloride
GC/MS	EPA 8260C	Bromobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Bromochloromethane
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	n-Butanol
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chlorobromomethane
GC/MS	EPA 8260C	2-Chloro-1,3-butadiene
GC/MS	EPA 8260C	Chlorodibromomethane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Allyl chloride
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Cyclohexanone
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260C	1,2-Dibromoethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	cis-1,2-Dichloroethene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,2-Dichloroethene (total)
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2-Dichloro-1,1,2,2-tetrafluoroethane
GC/MS	EPA 8260C	Dimethyl disulfide
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	Ethyl acetate
GC/MS	EPA 8260C	Ethylbenzene
GC/MS	EPA 8260C	Ethyl ether
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Freon 113
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	n-Hexane
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	p-Isopropyltoluene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methyl butyl ketone
GC/MS	EPA 8260C	Methylcyclohexane
GC/MS	EPA 8260C	Dichloromethane
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	4-Methyl-2-pentanone
GC/MS	EPA 8260C	MTBE
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	2-Nitropropane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Nonanal
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260C	Trichlorotrifluoroethane
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	m-Xylene & p-Xylene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Xylenes (total)
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite (total)
GC/MS	EPA 8270D	Atrazine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Azobenzene
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo(a)anthracene
GC/MS	EPA 8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270D	Benzoic acid
GC/MS	EPA 8270D	Benzo(ghi)perylene
GC/MS	EPA 8270D	Benzo(a)pyrene
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	1,1'-Biphenyl
GC/MS	EPA 8270D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270D	bis(2-Chloroisopropyl) ether
GC/MS	EPA 8270D	bis(2-Ethylhexyl) phthalate
GC/MS	EPA 8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270D	n-Butylbenzenesulfonamide
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	Chlorobenzilate
GC/MS	EPA 8270D	p-Chlorobenzilate
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols (total)
GC/MS	EPA 8270D	Cyclohexanol
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	1,2-Dichlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dichlorophenol
GC/MS	EPA 8270D	Diethyl phthalate
GC/MS	EPA 8270D	O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	Dimethylformamide
GC/MS	EPA 8270D	alpha,alpha-Dimethylphenethylamine
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dinitrobenzene
GC/MS	EPA 8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	Dinoseb
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methacrylate
GC/MS	EPA 8270D	Ethyl methanesulfonate
GC/MS	EPA 8270D	Famphur
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorobutadiene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloro-1,3-cyclopentadiene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D	Isodrin
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	Isosafrole
GC/MS	EPA 8270D	Kepone
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	2-Methylbenzenamine
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	4,4'-Methylenebis(2-chloroaniline)
GC/MS	EPA 8270D	Methyl methacrylate
GC/MS	EPA 8270D	Methyl methanesulfonate
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	Methyl parathion
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	3-Methylphenol & 4-Methylphenol
GC/MS	EPA 8270D	2-Methylphenol, 3-methylphenol and 4-methylphenol
GC/MS	EPA 8270D	Methylphenols (total)
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Naphthylamine
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	N-Nitrosodi-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine
GC/MS	EPA 8270D	5-Nitro-o-toluidine
GC/MS	EPA 8270D	2,2'-oxybis(1-Chloropropane)
GC/MS	EPA 8270D	Parathion
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachloroethane
GC/MS	EPA 8270D	Pentachloronitrobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	2-Picoline
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	Tetraethyldithiopyrophosphate (Sulfotepp)
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270D	o-Toluidine
GC/MS	EPA 8270D	Tributyl phosphate
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	Tris(2-chloroethyl)phosphate
GC/MS	EPA 8270D	1-Methyl naphthalene
GC-ECD	EPA 8081B	Aldrin

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081B	alpha-BHC
GC-ECD	EPA 8081B	beta-BHC
GC-ECD	EPA 8081B	delta-BHC
GC-ECD	EPA 8081B	gamma-BHC (Lindane)
GC-ECD	EPA 8081B	alpha-Chlordane
GC-ECD	EPA 8081B	gamma-Chlordane
GC-ECD	EPA 8081B	Chlordane (technical)
GC-ECD	EPA 8081B	4,4'-DDD
GC-ECD	EPA 8081B	2,4'-DDD
GC-ECD	EPA 8081B	4,4'-DDE
GC-ECD	EPA 8081B	2,4'-DDE
GC-ECD	EPA 8081B	4,4'-DDT
GC-ECD	EPA 8081B	2,4'-DDT
GC-ECD	EPA 8081B	Dieldrin
GC-ECD	EPA 8081B	Endosulfan I
GC-ECD	EPA 8081B	Endosulfan II
GC-ECD	EPA 8081B	Endosulfan sulfate
GC-ECD	EPA 8081B	Endrin
GC-ECD	EPA 8081B	Endrin aldehyde
GC-ECD	EPA 8081B	Endrin ketone
GC-ECD	EPA 8081B	Heptachlor
GC-ECD	EPA 8081B	Heptachlor epoxide
GC-ECD	EPA 8081B	Methoxychlor
GC-ECD	EPA 8081B	Toxaphene
GC-ECD	EPA 8082A	Aroclor 1016
GC-ECD	EPA 8082A	Aroclor 1221
GC-ECD	EPA 8082A	Aroclor 1232
GC-ECD	EPA 8082A	Aroclor 1242
GC-ECD	EPA 8082A	Aroclor 1248
GC-ECD	EPA 8082A	Aroclor 1254
GC-ECD	EPA 8082A	Aroclor 1260
GC-ECD	EPA 8082A	Aroclor 1262
GC-ECD	EPA 8082A	Aroclor 1268
GC-ECD	EPA 8151A	2,4-D
GC-ECD	EPA 8151A	Dalapon
GC-ECD	EPA 8151A	2,4-DB

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8151A	Dicamba
GC-ECD	EPA 8151A	Dichlorprop
GC-ECD	EPA 8151A	Dinoseb
GC-ECD	EPA 8151A	MCPA
GC-ECD	EPA 8151A	MCPP
GC-ECD	EPA 8151A	4-Nitrophenol
GC-ECD	EPA 8151A	Pentachlorophenol
GC-ECD	EPA 8151A	2,4,5-TP (Silvex)
GC-ECD	EPA 8151A	2,4,5-T
LC/MS/MS	EPA 8321A	2,4-D
LC/MS/MS	EPA 8321A	Dalapon
LC/MS/MS	EPA 8321A	2,4-DB
LC/MS/MS	EPA 8321A	Dicamba
LC/MS/MS	EPA 8321A	Dichlorprop
LC/MS/MS	EPA 8321A	Dinoseb
LC/MS/MS	EPA 8321A	MCPA
LC/MS/MS	EPA 8321A	MCPP
LC/MS/MS	EPA 8321A	2,4,5-TP (Silvex)
LC/MS/MS	EPA 8321A	2,4,5-T
LC/MS/MS	EPA 8321A	2-Amino-4,6-dinitrotoluene
LC/MS/MS	EPA 8321A	4-Amino-2,6-dinitrotoluene
LC/MS/MS	EPA 8321A	3,5-Dinitroaniline
LC/MS/MS	EPA 8321A	1,3-Dinitrobenzene
LC/MS/MS	EPA 8321A	2,4-Dinitrotoluene
LC/MS/MS	EPA 8321A	2,6-Dinitrotoluene
LC/MS/MS	EPA 8321A	DNX
LC/MS/MS	EPA 8321A	HMX
LC/MS/MS	EPA 8321A	HNAB
LC/MS/MS	EPA 8321A	HNS
LC/MS/MS	EPA 8321A	MNX
LC/MS/MS	EPA 8321A	Nitrobenzene
LC/MS/MS	EPA 8321A	Nitroglycerin
LC/MS/MS	EPA 8321A	4-Nitrotoluene
LC/MS/MS	EPA 8321A	3-Nitrotoluene
LC/MS/MS	EPA 8321A	2-Nitrotoluene
LC/MS/MS	EPA 8321A	PETN

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 8321A	RDX
LC/MS/MS	EPA 8321A	TATB
LC/MS/MS	EPA 8321A	Tetryl
LC/MS/MS	EPA 8321A	TNX
LC/MS/MS	EPA 8321A	1,3,5-Trinitrobenzene
LC/MS/MS	EPA 8321A	2,4,6-Trinitrotoluene
LC/MS/MS	EPA 8321A	Tris (o-cresyl) Phosphate
LC/MS/MS	EPA 8321A	2,4-diamino-6-nitrotoluene
LC/MS/MS	EPA 8321A	2,6-diamino-4-nitrotoluene
HPLC	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC	EPA 8330B	1,3-Dinitrobenzene
HPLC	EPA 8330B	2,4-Dinitrotoluene
HPLC	EPA 8330B	2,6-Dinitrotoluene
HPLC	EPA 8330B	HMX
HPLC	EPA 8330B	HNAB
HPLC	EPA 8330B	HNS
HPLC	EPA 8330B	Nitrobenzene
HPLC	EPA 8330B	Nitroglycerin
HPLC	EPA 8330B	2-Nitrotoluene
HPLC	EPA 8330B	3-Nitrotoluene
HPLC	EPA 8330B	4-Nitrotoluene
HPLC	EPA 8330B	PETN
HPLC	EPA 8330B	RDX
HPLC	EPA 8330B	TATB
HPLC	EPA 8330B	Tetryl
HPLC	EPA 8330B	MNX
HPLC	EPA 8330B	DNX
HPLC	EPA 8330B	TNX
HPLC	EPA 8330B	1,3,5-Trinitrobenzene
HPLC	EPA 8330B	2,4,6-Trinitrotoluene
GC/MS	EPA 8270D SIM	Acenaphthene
GC/MS	EPA 8270D SIM	Acenaphthylene
GC/MS	EPA 8270D SIM	Anthracene
GC/MS	EPA 8270D SIM	Benzo(a)anthracene
GC/MS	EPA 8270D SIM	Benzo(b)fluoranthene

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270D SIM	Benzo(ghi)perylene
GC/MS	EPA 8270D SIM	Benzo(a)pyrene
GC/MS	EPA 8270D SIM	Chrysene
GC/MS	EPA 8270D SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D SIM	Fluoranthene
GC/MS	EPA 8270D SIM	Fluorene
GC/MS	EPA 8270D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D SIM	Naphthalene
GC/MS	EPA 8270D SIM	Phenanthrene
GC/MS	EPA 8270D SIM	Pyrene
GC/MS	EPA 8260C SIM	1,4- dioxane
GC-FID	EPA 8015B	Diesel Range Organics
GC-FID	EPA 8015B	Motor Oil Range Organics
GC-FID	EPA 8015B	TPH (as Diesel)
GC-FID	EPA 8015B	Gasoline Range Organics
GC-FID	EPA 8015B	Ethanol
GC-FID	EPA 8015B	Methanol
GC-FID	EPA 8015B	Ethylene glycol
GC-FID	EPA 8015B	Propylene glycol
LC/MS/MS	EPA 6850	Perchlorate
ICP-MS	EPA 6020A	Aluminum
ICP-MS	EPA 6020A	Antimony
ICP-MS	EPA 6020A	Arsenic
ICP-MS	EPA 6020A	Barium
ICP-MS	EPA 6020A	Beryllium
ICP-MS	EPA 6020A	Bismuth
ICP-MS	EPA 6020A	Boron
ICP-MS	EPA 6020A	Cadmium
ICP-MS	EPA 6020A	Calcium
ICP-MS	EPA 6020A	Cerium
ICP-MS	EPA 6020A	Cesium
ICP-MS	EPA 6020A	Chromium
ICP-MS	EPA 6020A	Cobalt
ICP-MS	EPA 6020A	Copper
ICP-MS	EPA 6020A	Hafnium

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020A	Iron
ICP-MS	EPA 6020A	Lanthanum
ICP-MS	EPA 6020A	Lead
ICP-MS	EPA 6020A	Lithium
ICP-MS	EPA 6020A	Magnesium
ICP-MS	EPA 6020A	Manganese
ICP-MS	EPA 6020A	Molybdenum
ICP-MS	EPA 6020A	Neodymium
ICP-MS	EPA 6020A	Nickel
ICP-MS	EPA 6020A	Niobium
ICP-MS	EPA 6020A	Palladium
ICP-MS	EPA 6020A	Phosphorus
ICP-MS	EPA 6020A	Platinum
ICP-MS	EPA 6020A	Potassium
ICP-MS	EPA 6020A	Praseodymium
ICP-MS	EPA 6020A	Rhodium
ICP-MS	EPA 6020A	Ruthenium
ICP-MS	EPA 6020A	Samarium
ICP-MS	EPA 6020A	Selenium
ICP-MS	EPA 6020A	Silicon
ICP-MS	EPA 6020A	Silver
ICP-MS	EPA 6020A	Sodium
ICP-MS	EPA 6020A	Strontium
ICP-MS	EPA 6020A	Sulfur
ICP-MS	EPA 6020A	Tantalum
ICP-MS	EPA 6020A	Technetium-99
ICP-MS	EPA 6020A	Tellurium
ICP-MS	EPA 6020A	Thallium
ICP-MS	EPA 6020A	Thorium
ICP-MS	EPA 6020A	Tin
ICP-MS	EPA 6020A	Titanium
ICP-MS	EPA 6020A	Tungsten
ICP-MS	EPA 6020A	Uranium
ICP-MS	EPA 6020A	Uranium 233
ICP-MS	EPA 6020A	Uranium 234
ICP-MS	EPA 6020A	Uranium 235

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020A	Uranium 236
ICP-MS	EPA 6020A	Uranium 238
ICP-MS	EPA 6020A	Vanadium
ICP-MS	EPA 6020A	Yttrium
ICP-MS	EPA 6020A	Zinc
ICP-MS	EPA 6020A	Zirconium
CVAA	EPA 7471B	Mercury
Colormetric	EPA 9010C EPA 9012B	Cyanide
Ion Chromatrography	EPA 300.0 EPA 9056A	Bromide
Ion Chromatrography	EPA 300.0 EPA 9056A	Chloride
Ion Chromatrography	EPA 300.0 EPA 9056A	Fluoride
Ion Chromatrography	EPA 300.0 EPA 9056A	Nitrate
Ion Chromatrography	EPA 300.0 EPA 9056A	Nitrite
Ion Chromatrography	EPA 300.0 EPA 9056A	Sulfate
Ion Chromatrography	EPA 300.0 EPA 9056A	Ortho-phosph
Ion Chromatrography	EPA 300.0 EPA 9056A	Iodide
Ion Chromatrography	EPA 314.0	Perchlorate
Gravimetric	SM 2540B SM 2540C SM 2540D	Solids
Probe	EPA 9040C EPA 9045D EPA 150.1	pH
Titration	SM 2320B EPA 310.1	Alkalinity
Titration	EPA 9030	Sulfide
Penske-Martin	EPA1010A	Ignitability
Colormetric	EPA 353.1	nitrate/Nitrite
Colormetric	EPA 350.1	Ammonia
Colormetric	EPA 351.2	TKN
TOC Analyzer	EPA 9060A	TOC

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Colormetric	EPA 7196A	Hex Chromium
Gravimetric	EPA 1664A	Oil & Grease
Gravimetric	EPA 1664A	TPH
Probe	EPA 9050A	Conductivity
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 / DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium-137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Calcium-45
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese-56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 83
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Uranium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	Laboratory SOP ST-RC-0210	Polonium-210
Liquid Scintillation Counter	Eichrom Technologies FEW01	Iron-55
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129
Preparation	**Method**	**Type**
Organic Extraction & Sample Prep	EPA 3500C	Organic Extraction & Sample Prep
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Organic Cleanup	EPA 3600A	Cleanup for Organic extracts
Organic prep/analysis	EPA 8000C	Determinative Chromatographic Separations
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Acid Digestion (solids)	EPA 3050B	Acid Digestion for Metals of Sedimtent/Soils
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile Samples
Closed System Purge & Trap and Extraction for Volatiles	EPA 5035A	Closed System Purge & Trap and Extraction for Volatiles
Sep Funnel Liquid-Liquid Extraction	EPA 3510C	Sep Funnel Liquid-Liquid Extraction
Ultrasonic Extraction	EPA 3550C	Ultrasonic Extraction Organic Soils
Solid Phase Extraction	EPA 3535A	Solid Phase Extraction
Acid Clean-up	EPA 3665A	Acid Clean Up for PCBs

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Solid and Chemical Materials		
Preparation	**Method**	**Type**
Florisil Cleanup	EPA 3620C	Florisil Cleanup
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup
Waste Dilution	EPA 3585	Waste Dilution Volatile Organics
Waste Dilution	EPA 3580A	Waste Dilution SemiVolatile Organics
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction
Alkaline Digestion	EPA 3060A	Alkaline Digestion for Hexavalent Chromium

Notes:

1) This laboratory offers commercial testing service.

Approved by: _____ Date: January 2, 2015

R. Douglas Leonard
Chief Technical Officer

Re-Issued: 5/31/13 Revised: 6/17/13 Revised: 12/5/13 Revised: 12/9/13 Revised: 12/16/13
Revised: 5/23/14 Revised: 1/2/15

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA LABORATORIES SACRAMENTO

880 Riverside Parkway
West Sacramento, CA 95605
Karla Buechler Phone: 916 373 5600

ENVIRONMENTAL

Valid To: January 31, 2016 Certificate Number: 2928.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality Systems Manual for Environmental Laboratories) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Inductively Coupled Plasma (ICP), ICP-Mass Spectroscopy, Atomic Absorption Spectroscopy (flame), Gas Chromatography(GC), GC- Mass Spectroscopy, High Resolution Gas Chromatography/High Resolution Mass Spectroscopy, Liquid Chromatography(LC),LC- Mass Spectroscopy, Ion Chromatography, Spectrophotometry, Misc.- Electronic Probes

Parameter/Analyte	**Program: Solid/Hazardous Waste Matrix: Non-Potable Water**	**Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials**	**Air**
Metals			
Aluminum	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Antimony	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Arsenic	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Barium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Beryllium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Boron	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	-------
Cadmium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Calcium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Chromium (Total)	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Chromium (Hexavalent)	EPA 7196A	EPA 7196A	-------
Cobalt	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Copper	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A

5301 Buckeystown Pike, Suite 350 | Frederick, Maryland 21704-8373 | Phone: 301 644 3248 | Fax: 301 662 2974 | www.A2LA.org

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Iron	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Lead	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Magnesium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Manganese	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Mercury	EPA 7470A	EPA 7471A	-------
Molybdenum	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Nickel	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Phosphorus	EPA 6020/6020A	EPA 6020/6020A	-------
Potassium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Selenium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Silica	EPA 6010B/6010C	-------	-------
Silicon	EPA 6010B/6010C	-------	-------
Silver	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Sodium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Strontium	EPA 6020/6020A	EPA 6020/6020A	-------
Thallium	EPA 6010B/6020 6010C/6020A	EPA 6010B/6020 6010C/6020A	EPA 6020/6020A
Thallium	EPA 6010B/6020	EPA 6010B/6020	EPA 6020/6020A
Tin	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	-------
Titanium	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	-------
Uranium	EPA 6020/6020A	EPA 6020/6020A	-------
Vanadium	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
Zinc	EPA 6010B/6010C/6020//6020A	EPA 6010B/6010C/6020//6020A	EPA 6020/6020A
<u>Nutrients</u>			
Nitrate	EPA 353.2/9056A/300.0	EPA 353.2/ 9056A/300.0	-------
Nitrate-nitrite	EPA 353.2	EPA 353.2	-------
Nitrite	EPA 353.2/9056A/300.0	EPA 353.2/9056A/300.0	-------
Orthophosphate	EPA 9056A/300.0	EPA 9056A/300.0	-------
<u>Wet Chemistry</u>			
Alkalinity	SM 2320B (1997)	-------	-------
Chemical Oxygen Demand	EPA 410.4	-------	-------
Oil and Grease	EPA 1664A/9070	EPA 1664A/9071	-------
Nitrocellulose	WS-WC-0050	WS-WC-0050	-------
Perchlorate	EPA 6850	EPA 6850	-------
pH	EPA 9040B/9040C	EPA 9040B/9040C	-------
Bromide	EPA 9056A/300.0	EPA 9056A/300.0	-------
Chloride	EPA 9056A/300.0	EPA 9056A/300.0	-------
Fluoride	EPA 9056A/300.0	EPA 9056A/300.0	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Sulfate	EPA 9056A/300.0	EPA 9056A/300.0	-------
Solids, Total	SM2540B (1997)	-------	-------
Solids, Total Suspended	SM2540D (1997)	-------	-------
Solids, Total Dissolved	SM2540C (1997)	-------	-------
%Moisture	-------	ASTM D2216	
TSP (Total Suspended Particulate)	-------	-------	40CFR Part 50 App B
PM10	-------	-------	40CFR Part 50 App J
Hazardous Waste Characteristics			
TCLP Extractables	-------	EPA 1311	-------
TCLP Inorganics	-------	EPA 1311	-------
Purgeable Organics			
(volatiles)			
1,1,1,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------
1,1,1-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1,2,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1,2-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1-2-Trichloro-1,2-2-trifluorethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,1-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	-------
1,2,3-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2,3-Trichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2,4-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2,4-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2-Dibromo-3-chloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------
1,2-Dibromoethane	EPA 8260B/8260C	EPA 8260B/8260C	-------
1,2-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,3,5-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,3-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,3-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------
1,4-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,4-Dioxane	-------	-------	TO14A/TO15

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
1-Chlorocyclohex ane	EPA 8260B/8260C	EPA 8260B/8260C	-------
2,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------
2-Butanone (MEK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
2-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
2-Hexanone (MBK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
2-Methyl-2-propanol (tert-Butyl Alcohol,(TBA)	-------	-------	TO14A/TO15
4-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	-------
4-Ethyltoluene	-------	-------	TO14A/TO15
4-Isopropyltoluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
4-Methyl-2-pentanone (MIBK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Acetone	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Acrolein	-------	-------	TO14A/TO15
Allyl Chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Alpha Methyl Styrene	-------	-------	TO14A/TO15
Benzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15; TO3
Benzyl chloride	-------	-------	TO14A/TO15
Bromobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------
Bromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------
Bromodichloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Bromoform	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Bromomethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Butadiene (1,3-Butadiene)	-------	-------	TO14A/TO15
Butane	-------	-------	TO14A/TO15
Carbon disulfide	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15; EPA 15/16
Carbon tetrachloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Chlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Chlorodifluoromethane	-------	-------	TO14A/TO15
Chloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Chloroform	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Chloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
cis-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
cis-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Cyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dibromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dibromomethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dichlorodifluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dimethyl Disulfide	WS-MS-0003	WS-MS-0003	EPA 15/16
Ethyl acetate	-------	-------	TO14A/TO15
Ethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15; TO3
Ethylene Dibromide (EDB)	-------	-------	TO14A/TO15

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Ethylmethacrylate	EPA 8260B/8260C	EPA 8260B/8260C	-------
Hexachlorobutadiene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Hexane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Iodomethane	EPA 8260B/8260C	EPA 8260B/8260C	-------
Isobutanol	EPA 8260B/8260C	EPA 8260B/8260C	-------
Isooctane (2,2,4-Trimethylpentane)	-------	-------	TO14A/TO15
Isopropyl alcohol	-------	-------	TO14A/TO15
Isopropylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
m & p xylene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15; TO3
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15; TO3
Methylene chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Naphthalene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
n-Butanol	-------	-------	TO14A/TO15
n-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
n-Heptane	-------	-------	TO14A/TO15
n-Nonane	-------	-------	TO14A/TO15
n-Octane	-------	-------	TO14A/TO15
n-Propylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Oxygenates	EPA 8260B/8260C	EPA 8260B/8260C	-------
o-xylene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15; TO3
Pentane	-------	-------	TO14A/TO15
Propene	-------	-------	TO14A/TO15
sec-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Styrene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
T-1,4-Dichloro-2-Butene	EPA 8260B/8260C	EPA 8260B/8260C	-------
tert-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Tetrachloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Tetrahydrofuran	-------	-------	TO14A/TO15
Toluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15; TO3
trans-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
trans-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Trichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Trichlorofluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Vinyl acetate	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Vinyl bromide	-------	-------	TO14A/TO15
Vinyl chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Xylenes, Total	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Hydrogen sulfide	-------	-------	EPA 15/16
Carbonyl sulfide	-------	-------	EPA 15/16

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Methanethiol (Methyl mercaptan)	-------	-------	EPA 15/16
Ethanethiol (Ethyl mercaptan)	-------	-------	EPA 15/16
Dimethyl sulfide	-------	-------	EPA 15/16
TPH as Gasoline	-------	-------	TO3
Carbon Dioxide	-------	-------	ASTM1946D /3C
Nitrogen	-------	-------	ASTM1946D /3C
Oxygen	-------	-------	ASTM1946D /3C
Helium	-------	-------	ASTM1946D /3C
Hydrogen	-------	-------	ASTM1946D /3C
Methane	-------	-------	ASTM1946D /3C
Gasoline Range Organics (GRO)	EPA 8260B/AK101MS	EPA 8260B/AK101MS	TO14A/TO15
TPH as Gasoline			TO3
Extractable Organics			
(semivolatiles)			
1,2,4,5-Tetrachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
1,2,4-Trichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
1,2-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
1,2-Diphenylhydrazine (as Azobenzene)	EPA 8270C/8270D	EPA 8270C/8270D	-------
1,3-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
1,3-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
1,4-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
1-Methylnaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,3,4,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,4,5-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,4,6-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,4-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,4-Dimethylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,4-Dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,4-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,6-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2,6-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	-------
2-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------
2-Chlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2-Methylnaphthalene	EPA 8270C/8270D/ WS-MS-0008 8270C-SIM/8270D-SIM	EPA 8270C/8270D/ WS-MS-0008 8270C-SIM/8270D-SIM	WS-MS-0006
2-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
2-Naphthylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
2-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------
2-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
3&4-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
3,3'-Dichlorobenzidine	EPA 8270C/8270D	EPA 8270C/8270D	-------
3-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------
4,6-Dinitro-2-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
4-Bromophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	-------
4-Chloro-3-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
4-Chloroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------
4-Chlorophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	-------
4-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------
4-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
Acenaphthene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Acenaphthylene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Aniline	EPA 8270C/8270D	EPA 8270C/8270D	-------
Anthracene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Benzo(a)anthracene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Benzo(a)pyrene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Benzo(b)fluoranthene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Benzo(g,h,i)perylene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Benzo(k)fluoranthene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Benzoic acid	EPA 8270C/8270D	EPA 8270C/8270D	-------
Benzyl alcohol	EPA 8270C/8270D	EPA 8270C/8270D	-------
Benzyl butyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------
Biphenyl	EPA 8270C/8270D	EPA 8270C/8270D	-------
Bis(2-chloroethoxy) Methane	EPA 8270C/8270D	EPA 8270C/8270D	-------
Bis(2-chloroethyl) ether	EPA 8270C/8270D	EPA 8270C/8270D	-------
Bis(2-chloroisopropyl) ether	EPA 8270C/8270D	EPA 8270C/8270D	-------
Carbazole	EPA 8270C/8270D	EPA 8270C/8270D	-------
Chrysene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Bis (2-ethylhexyl) phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------
Diallate	EPA 8270C/8270D	EPA 8270C/8270D	-------
Dibenz(a,h)anthracene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Dibenzofuran	EPA 8270C/8270D	EPA 8270C/8270D	-------
Diethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------
Dimethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------
Di-n-butyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------
Di-n-octyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------
Fluoranthene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Fluorene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Hexachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
Hexachlorobutadiene	EPA 8270C/8270D	EPA 8270C/8270D	-------
Hexachlorocyclopentadiene	EPA 8270C/8270D	EPA 8270C/8270D	-------
Hexachloroethane	EPA 8270C/8270D	EPA 8270C/8270D	-------
Indeno(1,2,3-c,d)pyrene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Isophorone	EPA 8270C/8270D	EPA 8270C/8270D	-------
Naphthalene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Nitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------
n-Nitrosodimethylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------
n-Nitrosodi-n-propylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------
n-Nitrosodiphenylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------
Pentachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
Phenanthrene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
Phenol	EPA 8270C/8270D	EPA 8270C/8270D	-------
Pyrene	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	EPA 8270C/8270D/8270C-SIM/8270D-SIM/WS-MS-0008	WS-MS-0006
1,4-Dioxane	WS-MS-0011	WS-MS-0011	-------
Diesel Range Organics (DRO)	EPA 8015B/8015D/AK102	EPA 8015B/8015D/AK102	-------
Residual Range Organics	AK103	AK103	-------
Dioxins			
2,3,7,8-TeCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,7,8-PeCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,4,7,8-HxCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,6,7,8-HxCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,7,8,9-HxCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
1,2,3,4,6,7,8-HpCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
OCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
2,3,7,8-TeCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,7,8-PeCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
2,3,4,7,8-PeCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,4,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,6,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,7,8,9-HxCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
2,3,4,6,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,4,6,7,8-HpCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
1,2,3,4,7,8,9-HpCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
OCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total TCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total PeCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total HxCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total HeptaCDD	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total TCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total PeCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total HxCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Total HpCDF	EPA 8280A/8280B/8290/8290A/1613B	EPA 8280A/8280B/ 8290/8290A/1613B	-------
Chemical Warfare Degradates			
1,4-Dithiane	WS-MS-0003	WS-MS-0003	-------
Benzothiazole	WS-MS-0003	WS-MS-0003	-------
p-Chlorophenyl methylsulfide	WS-MS-0003	WS-MS-0003	-------
p-Chlorophenyl methylsulfoxide	WS-MS-0003	WS-MS-0003	-------
p-Chlorophenyl methylsulfone	WS-MS-0003	WS-MS-0003	-------
Chloropicrin	WS-MS-0003	WS-MS-0003	-------
Acetophenone	WS-MS-0003	WS-MS-0003	-------
2-Chloroacetophenone	WS-MS-0003	WS-MS-0003	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
1,4-Oxathiane	WS-MS-0003	WS-MS-0003	-------
Dimethyl Disulfide	WS-MS-0003	WS-MS-0003	EPA 15/16
Diisopropylmethylphosphate (DIMP)	WS-LC-0004	WS-LC-0004	-------
Dimethylmethylphosphonate (DMMP)	WS-LC-0004	WS-LC-0004	-------
Ethyl methylphosphonic acid (EMPA)	WS-LC-0004	WS-LC-0004	-------
Isopropyl methylphosphonic acid (IMPA)	WS-LC-0004	WS-LC-0004	-------
Methylphosphonic acid (MPA)	WS-LC-0004	WS-LC-0004	-------
Thiodiglycol (2,2'-Thiodiethanol) (TDG)	WS-LC-0004	WS-LC-0004	-------
Nitroaromatics			
2-Amino-4,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------
4-Amino-2,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------
3,5-Dinitroaniline	EPA 8330B	EPA 8330B	-------
1,3-Dinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	-------
2,4-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------
2,6-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------
Glycerol trinitrate (Nitroglycerin)	EPA 8330B	EPA 8330B	-------
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	EPA 8330A/8330B	EPA 8330A/8330B	-------
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330A/8330B	EPA 8330A/8330B	-------
Nitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	-------
2-Nitrotoluene (o-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	-------
3-Nitrotoluene (m-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	-------
4-Nitrotoluene (p-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	-------
Octahydro-1,3,5,7-tetranitro1,3,5,7-tetracine (Octogen)	EPA 8330A/8330B	EPA 8330A/8330B	-------
Picric acid	EPA 8330B	EPA 8330B	-------
Pentaerythritol tetranitrate	EPA 8330B	EPA 8330B	-------
1,3,5-Trinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	-------
2,4,6-Trinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------
Hexahydro-1,3-dinitroso-5-nitro-1,3,5,triazine (DNX)	EPA 8330A/8330B	EPA 8330A/8330B	-------
Hexahydro-1,3,5-trinitroso-1,3,5-triazine (TNX)	EPA 8330A/8330B	EPA 8330A/8330B	-------
1-Nitroso-3,5-dinitro-1,3,5-triazacyclohexane (MNX)	EPA 8330A/8330B	EPA 8330A/8330B	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Nitroguanidine	WS-LC-0010	WS-LC-0010	-------
Nitrosamines			
N-Nitrosodimethyl amine (NDMA)	WS-MS-0012	WS-MS-0012	-------
Perfluoro Compounds			
Perfluorooctanoic acid	WS-LC-0025	WS-LC-0025	-------
Perfluorooctane sulfonate	WS-LC-0025	WS-LC-0025	-------
Perfluorobutyric acid (PFBA)	WS-LC-0025	-------	-------
Perfluoropentanoic acid (PFPA)	WS-LC-0025	-------	-------
Perfluorohexanoic acid (PFHxA)	WS-LC-0025	-------	-------
Perfluoroheptanoic acid (PFHpA)	WS-LC-0025	WS-LC-0025	-------
Perfluorononanoic acid (PFNA)	WS-LC-0025	WS-LC-0025	-------
Perfluorodecanoic acid (PFDA)	WS-LC-0025	-------	-------
Perfluoroundecanoic acid (PFUDA	WS-LC-0025	-------	-------
Perfluorododecanoic acid (PFDoDA)	WS-LC-0025	-------	-------
Perfluorobutane Sulfonate (PFBS)	WS-LC-0025	WS-LC-0025	-------
Perfluorohexane Sulfonate (PFHxS)	WS-LC-0025	WS-LC-0025	-------
Perfluoroheptane Sulfonate (PFHpS)	WS-LC-0025	-------	-------
Perfluorodecane Sulfonate (PFDS)	WS-LC-0025	-------	-------
Pesticides/PCBs			
Aldrin	EPA 8081A/8081B	EPA 8081A/8081B	-------
a-BHC	EPA 8081A/8081B	EPA 8081A/8081B	-------
b-BHC	EPA 8081A/8081B	EPA 8081A/8081B	-------
d-BHC	EPA 8081A/8081B	EPA 8081A/8081B	-------
g-BHC (Lindane)	EPA 8081A/8081B	EPA 8081A/8081B	-------
a-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	-------
g-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	-------
4,4'-DDD	EPA 8081A/8081B	EPA 8081A/8081B	-------
4,4'-DDE	EPA 8081A/8081B	EPA 8081A/8081B	-------
4,4'-DDT	EPA 8081A/8081B	EPA 8081A/8081B	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Dieldrin	EPA 8081A/8081B	EPA 8081A/8081B	-------
Endosulfan I	EPA 8081A/8081B	EPA 8081A/8081B	-------
Endosulfan II	EPA 8081A/8081B	EPA 8081A/8081B	-------
Endosulfan sulfate	EPA 8081A/8081B	EPA 8081A/8081B	-------
Endrin	EPA 8081A/8081B	EPA 8081A/8081B	-------
Endrin aldehyde	EPA 8081A/8081B	EPA 8081A/8081B	-------
Endrin ketone	EPA 8081A/8081B	EPA 8081A/8081B	-------
Heptachlor	EPA 8081A/8081B	EPA 8081A/8081B	-------
Heptachlor epoxide	EPA 8081A/8081B	EPA 8081A/8081B	-------
Methoxychlor	EPA 8081A/8081B	EPA 8081A/8081B	-------
Toxaphene	EPA 8081A/8081B	EPA 8081A/8081B	-------
Chlordane (technical)	EPA 8081A/8081B	EPA 8081A/8081B	-------
PCB (Aroclors)			
PCB-1016	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1221	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1232	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1242	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1248	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1254	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1260	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1262	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1268	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB (congeners) – removed references to BZ and IUPAC numbers.			
PCB 1	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 2	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 3	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 4	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 5	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 6	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 7	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 8	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 9	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 10	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 11	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 12	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 13	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
PCB 14	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 15	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 16	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 17	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 18	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 19	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 20	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 21	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 22	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 23	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 24	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 25	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 26	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 27	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 28	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 29	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 30	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 32	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 31	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 33	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 34	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 35	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 36	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 37	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 38	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 39	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 40	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 41	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 42	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 43	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 44	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 45	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 46	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 47	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 48	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 49	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 50	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 51	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 52	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 53	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 54	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 55	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
PCB 56	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 57	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 58	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 59	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 60	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 61	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 62	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 63	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 64	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 65	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 66	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 67	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 68	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 69	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 70	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 71	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 72	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 73	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 74	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 75	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 76	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 77	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 78	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 79	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 80	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 81	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 82	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 83	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 84	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 85	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 86	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 87	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 88	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 89	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 90	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 91	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 92	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 93	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 94	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 95	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 96	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 97	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
PCB 98	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 99	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 100	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 101	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 102	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 103	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 104	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 105	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 106	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 107	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 108	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 109	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 110	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 111	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 112	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 113	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 114	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 115	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 116	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 117	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 118	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 119	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 120	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 121	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 122	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 123	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 124	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 125	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 126	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 127	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 128	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 129	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 130	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 131	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 132	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 133	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 134	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 135	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 136	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 137	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 138	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 139	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
PCB 140	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 141	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 142	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 143	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 144	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 145	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 146	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 147	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 148	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 149	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 150	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 151	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 152	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 153	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 154	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 155	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 156	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 157	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 158	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 159	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 160	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 161	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 162	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 163	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 164	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 165	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 166	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 167	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 168	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 169	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 170	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 171	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 172	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 173	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 174	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 175	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 176	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 177	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 178	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 179	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 180	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 181	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
PCB 182	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 183	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 184	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 185	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 186	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 187	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 188	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 189	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 190	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 191	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 192	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 193	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 194	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 195	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 196	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 197	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 198	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 199	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 200	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 201	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 202	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 203	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 204	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 205	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 206	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 207	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 208	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
PCB 209	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------
Metals Digestion			
Acid Digestion Total Rcoverable or Dissolved Metals	EPA 3005A	-------	-------
Acid Digestion for Total Metals	EPA 3010A	-------	-------
Acid Digestion of Sediments, Sludges and Soils	-------	EPA 3050B	EPA 3050B
Organic Preparation Methods			
Separatory Funnel Liquid-Liquid Extraction	-------	EPA 3510C	-------
Ultrasonic Extraction	-------	EPA 3550B, EPA 3550C	-------
Waste Dilution	EPA 3580A	EPA 3580A	-------

Parameter/Analyte	Program: Solid/Hazardous Waste Matrix: Non-Potable Water	Program: Solid/Hazardous Waste Matrix: Solid/Chemical Materials	Air
Solid-Phase Extraction	EPA 3535A		-------
Volatiles Purge and Trap	EPA 5030B, EPA 5030C	EPA 5030B	-------
Volatiles Purge and Trap for Solids	-------	EPA 5035, EPA 5035A	-------
Semivolatiles in Air	-------	-------	EPA 3542 TO-13
Chemical Warfare Degradates (in solid)	-------	WS-OP-0005	-------
Organic Cleanup Procedures			
Florisil Cleanup	EPA 3620B, EPA3620C	EPA 3620B, EPA3620C	EPA 3620B, EPA3620C
Sulfur Cleanup	EPA 3660A	EPA 3660A	EPA 3660A
Sulfuric Acid Cleanup	EPA 3665A	EPA 3665A	EPA 3665A
Silica Gel Cleanup	EPA 3630C	EPA 3630C	-------

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World Class Accreditation

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A2LA has accredited

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West Sacramento, CA

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2009 TNI Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.0 of the DoD Quality System Manual for Environmental Laboratories (QSM); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (*refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009*).

AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
CORPORATE SEAL 1978
DISTRICT OF COLUMBIA
A2LA

Presented this 14th day of May 2014.

President & CEO
For the Accreditation Council
Certificate Number 2928.01
Valid to January 31, 2016

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

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CALIFORNIA STATE
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing

TestAmerica Sacramento

880 Riverside Parkway
West Sacramento, CA 95605
Phone: (916) 373-5600

Certificate No.: 2897
Renew Date: 1/31/2016
INTERIM

Field of Testing: 102 - Inorganic Chemistry of Drinking Water

102.045	001	Perchlorate	EPA 314.0
102.047	001	Perchlorate	EPA 331.0

Field of Testing: 105 - Semi-volatile Organic Chemistry of Drinking Water

105.230	000	Dioxins	EPA 1613
105.230	001	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	EPA 1613

Field of Testing: 108 - Inorganic Chemistry of Wastewater

108.020	001	Conductivity	EPA 120.1
108.112	001	Boron	EPA 200.7
108.113	001	Boron	EPA 200.8
108.120	001	Bromide	EPA 300.0
108.120	002	Chloride	EPA 300.0
108.120	003	Fluoride	EPA 300.0
108.120	004	Nitrate	EPA 300.0
108.120	005	Nitrite	EPA 300.0
108.120	006	Nitrate-nitrite	EPA 300.0
108.120	007	Phosphate, Ortho	EPA 300.0
108.120	008	Sulfate	EPA 300.0
108.232	001	Nitrate-nitrite	EPA 353.2
108.232	002	Nitrite	EPA 353.2
108.323	001	Chemical Oxygen Demand	EPA 410.4
108.381	001	Oil and Grease	EPA 1664A
108.420	001	Hardness (calculation)	SM2340B
108.421	001	Hardness	SM2340C
108.430	001	Conductivity	SM2510B
108.440	001	Residue, Total	SM2540B
108.441	001	Residue, Filterable TDS	SM2540C
108.442	001	Residue, Non-filterable TSS	SM2540D
108.490	001	Hydrogen Ion (pH)	SM4500-H+ B
108.522	001	Nitrate-nitrite	SM4500-NO3 F
108.522	002	Nitrite	SM4500-NO3 F

Field of Testing: 109 - Toxic Chemical Elements of Wastewater

109.010	001	Aluminum	EPA 200.7
109.010	002	Antimony	EPA 200.7
109.010	003	Arsenic	EPA 200.7
109.010	004	Barium	EPA 200.7
109.010	005	Beryllium	EPA 200.7
109.010	006	Boron	EPA 200.7

109.010	007	Cadmium	EPA 200.7
109.010	009	Chromium	EPA 200.7
109.010	010	Cobalt	EPA 200.7
109.010	011	Copper	EPA 200.7
109.010	012	Iron	EPA 200.7
109.010	013	Lead	EPA 200.7
109.010	015	Manganese	EPA 200.7
109.010	016	Molybdenum	EPA 200.7
109.010	017	Nickel	EPA 200.7
109.010	019	Selenium	EPA 200.7
109.010	021	Silver	EPA 200.7
109.010	023	Thallium	EPA 200.7
109.010	024	Tin	EPA 200.7
109.010	025	Titanium	EPA 200.7
109.010	026	Vanadium	EPA 200.7
109.010	027	Zinc	EPA 200.7
109.020	001	Aluminum	EPA 200.8
109.020	002	Antimony	EPA 200.8
109.020	003	Arsenic	EPA 200.8
109.020	004	Barium	EPA 200.8
109.020	005	Beryllium	EPA 200.8
109.020	006	Cadmium	EPA 200.8
109.020	007	Chromium	EPA 200.8
109.020	008	Cobalt	EPA 200.8
109.020	009	Copper	EPA 200.8
109.020	010	Lead	EPA 200.8
109.020	011	Manganese	EPA 200.8
109.020	012	Molybdenum	EPA 200.8
109.020	013	Nickel	EPA 200.8
109.020	014	Selenium	EPA 200.8
109.020	015	Silver	EPA 200.8
109.020	016	Thallium	EPA 200.8
109.020	017	Vanadium	EPA 200.8
109.020	018	Zinc	EPA 200.8
109.020	021	Iron	EPA 200.8
109.020	022	Tin	EPA 200.8
109.020	023	Titanium	EPA 200.8
109.190	001	Mercury	EPA 245.1

Field of Testing: 110 - Volatile Organic Chemistry of Wastewater

110.040	000	Purgeable Organic Compounds	EPA 624
110.040	042	Oxygenates	EPA 624
110.040	043	Other Volatile Organics	EPA 624

Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater

111.100	000	Acid/base/neutral Organic Compounds	EPA 625
111.101	032	Polynuclear Aromatic Hydrocarbons	EPA 625
111.111	000	Dioxins	EPA 1613B

111.111	022	Total TCDF	EPA 1613B
111.111	023	Total PeCDF	EPA 1613B
111.111	024	Total HxCDF	EPA 1613B
111.111	025	Total HpCDF	EPA 1613B
111.170	000	Pesticides	EPA 608
111.273	001	Oil and Grease	EPA 1664A

Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste

114.010	001	Antimony	EPA 6010B
114.010	002	Arsenic	EPA 6010B
114.010	003	Barium	EPA 6010B
114.010	004	Beryllium	EPA 6010B
114.010	005	Cadmium	EPA 6010B
114.010	006	Chromium	EPA 6010B
114.010	007	Cobalt	EPA 6010B
114.010	008	Copper	EPA 6010B
114.010	009	Lead	EPA 6010B
114.010	010	Molybdenum	EPA 6010B
114.010	011	Nickel	EPA 6010B
114.010	012	Selenium	EPA 6010B
114.010	013	Silver	EPA 6010B
114.010	014	Thallium	EPA 6010B
114.010	015	Vanadium	EPA 6010B
114.010	016	Zinc	EPA 6010B
114.010	026	Silica	EPA 6010B
114.010	027	Sodium	EPA 6010B
114.020	001	Antimony	EPA 6020
114.020	002	Arsenic	EPA 6020
114.020	003	Barium	EPA 6020
114.020	004	Beryllium	EPA 6020
114.020	005	Cadmium	EPA 6020
114.020	006	Chromium	EPA 6020
114.020	007	Cobalt	EPA 6020
114.020	008	Copper	EPA 6020
114.020	009	Lead	EPA 6020
114.020	010	Molybdenum	EPA 6020
114.020	011	Nickel	EPA 6020
114.020	012	Selenium	EPA 6020
114.020	013	Silver	EPA 6020
114.020	014	Thallium	EPA 6020
114.020	015	Vanadium	EPA 6020
114.020	016	Zinc	EPA 6020
114.103	001	Chromium (VI)	EPA 7196A
114.140	001	Mercury	EPA 7470A
114.141	001	Mercury	EPA 7471A
114.240	001	Corrosivity - pH Determination	EPA 9040B
114.241	001	Corrosivity - pH Determination	EPA 9045C
114.250	001	Fluoride	EPA 9056

Field of Testing: 115 - Extraction Test of Hazardous Waste

115.021	001	TCLP Inorganics	EPA 1311
115.022	001	TCLP Extractables	EPA 1311
115.030	001	Waste Extraction Test (WET)	CCR Chapter11, Article 5, Appendix II
115.040	001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312

Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste

116.080	000	Volatile Organic Compounds	EPA 8260B
116.080	120	Oxygenates	EPA 8260B
116.100	001	Total Petroleum Hydrocarbons - Gasoline	LUFT GC/MS
116.100	002	Benzene	LUFT GC/MS
116.100	003	Toluene	LUFT GC/MS
116.100	004	Xylenes	LUFT GC/MS
116.100	005	Methyl tert-butyl Ether (MTBE)	LUFT GC/MS
116.100	010	BTEX and MTBE	LUFT GC/MS

Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.010	001	Diesel-range Total Petroleum Hydrocarbons	EPA 8015B
117.016	001	Diesel-range Total Petroleum Hydrocarbons	LUFT
117.110	000	Extractable Organics	EPA 8270C
117.120	000	Dioxins and Dibenzofurans	EPA 8280A
117.130	000	Dioxins and Dibenzofurans	EPA 8290
117.170	000	Nitroaromatics and Nitramines	EPA 8330
117.210	000	Pesticides	EPA 8081A
117.220	000	PCBs	EPA 8082

Attachment 3: Laboratory Standard Operating Procedures

To be included with the Final SAP on CD

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TestAmerica Denver

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Electronic Copy Only

Title: Dissolved Gases in Water
Method No. RSK-175

Approvals (Signature/Date):

Dennis Jonsrud Technical Specialist	9-30-14 Date	Adam Alban Health & Safety Manager / Coordinator	30 Sept 14 Date
Margaret S. Sleevi Quality Assurance Manager	9/30/14 Date	William S. Cicero Laboratory Director	9/30/14 Date

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1.0 Scope and Application

1.1. This document describes a procedure for the determination of methane, ethane, ethene, acetylene, propane, isobutylene, butane, and pentane dissolved in water using a static headspace autosampler connected to a gas chromatograph with dual flame ionization detectors (GC/FID). This SOP is based on the RSK-175 method described in EPA document EPA/600/J-89/186, 1994.

1.2. The most common application for this analysis is to assist in evaluating the biological activity in aquifers contaminated with petroleum hydrocarbons. It is also used by the fracking industry to establish baseline methane levels in groundwater monitoring.

1.3. Refer to Table 3 for a list of analytes and standard reporting limits.

2.0 Summary of Method

2.1 A water sample, with field blank and duplicate, is collected in a VOA vial with a screw cap fitted with a Teflon septum. The vial is completely filled so that there is no headspace. When the sample is received at the laboratory, 18 mL of the sample is transferred to a 22 mL serum vial with a crimp septum cap. The sample is loaded onto the headspace autosampler and analyzed by dual column GC/FID.

2.2 The instrument is calibrated using a minimum of 5 of the 7 calibration levels shown in Table 2. Sample results are calculated using the external standardization method.

2.3 Unless extrapolating results to conditions different than those used for analysis, this procedure does not require calculation of the concentration in the headspace using Henry's Law and then using this result to calculate the concentration in the aqueous sample. Instead of calibrating with gas standards, the calibration standards are prepared in water to mimic the actual field samples. Therefore, the calibration incorporates the equilibration between the aqueous and gaseous phases in the sample vial. Continuing calibration verification standards are analyzed immediately prior to sample analysis to ensure that the same temperature and pressure exist for both standards and samples.

3.0 Definitions

3.1 Henry's Law

Henry's Law states that the equilibrium value of the mole fraction of gas dissolved in a liquid is directly proportional to the partial pressure of the gas above the liquid surface, and is expressed as follows:

$$P_g = X_g \times K_h$$

Where:

P_g = partial pressure in atmospheres

X_g = mole fraction of dissolved gas, which given a liquid volume and the gram molecular weight per mole can be converted to a concentration

K_h = Henry's law constant in atmospheres, the value of the constant is dependent on temperature

Henry's Law is applicable at low concentrations and low partial pressures of gas at or below one atmosphere of pressure.

3.2 Static Headspace Autosampler

The term "static headspace" means that the sample is not stirred, mixed, or bubbled during analysis. The autosampler described in Section 6.2 includes a heating block that holds the 22 mL vials and ensures a constant 40°C (± 1°C) temperature during analysis.

3.3 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, "Quality Assurance Program," for definitions of general analytical and QA/QC terms.

4.0 Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.1 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Samples immediately following a high-level sample are re-analyzed if they exhibit any detection of target analytes. Typically carryover is not observed at sample concentrations that are within the calibration range.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual, and this document.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1 (as per the Environmental Health and Safety Manual), laboratory coat, and latex or nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Hydrogen Gas	Flammable High-Pressure Gas Asphyxiant	None established	Hydrogen gas can form explosive mixtures with air. May ignite if valve is opened to air. Burns with invisible flame. Inhalation effects are due to lack of oxygen. Moderate concentrations may cause headache, drowsiness, excitation, excess salivation, vomiting, and unconsciousness.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Supplies

6.1.1 Sample Collection Containers: 43 mL VOA vial with septum cap.

6.1.2 Sample Analysis Containers: 22 mL crimp cap vials. Each lot of 22 mL headspace vials must have the volume confirmed by tare weighing ten vials and then filling to capacity and reweighing. The data is to be entered in the spreadsheet found at R:\QA\Edit\FORMS\Support Equipment\Pipettes\(Att15) 22 mL vial Verification Sheet. The data is stored in a file G:\GcVoa\Hidden\pipette and syringe calibration checks\Vial (22ml) Calibration Verification Spreadsheet.

6.1.3 Columns:

6.1.3.1 Restek Rt-U-Bond, 30m x 0.32mm ID x 10um

6.1.3.2 Restek Rt-S-Bond, 30m x 0.32mm ID x 10um

6.1.3.3 Columns giving equivalent performance can also be used.

6.1.4 Syringes: 5 µL – 20.0 mL gas-tight syringes.

6.1.5 Several 12" transfer cannulas, 20ga.

6.1.6 Pressure relief valve (cannula or needle)

6.1.7 Source of N2 gas regulated to provide a sample flow through the cannula at 14-20 mL per minute.

6.1.8 Balance to weigh to nearest 0.01g.

6.1.9 Calibrated mechanical pipettes with pipette tips or Class A glass volumetric pipettes. Pipette calibration is checked in accordance with SOP DV-QA-0008.

6.2 Instrumentation

6.2.1 Instrumentation: Agilent Model 6890 GC with Dual FID detectors, or equivalent. Current instrument is instrument J.

6.2.2 Autosampler: Tekmar 7000 Headspace Autosampler

6.2.3 Data System: Chemstation for acquisition and Chrom for data processing, or equivalent.

6.3 Computer Software and Hardware

Refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Gas cylinders of ultrahigh purity helium, hydrogen, air, and nitrogen, > 99.999% pure.

7.2 Standards Verification

All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.3 Gas Standards:

7.3.1 Expiration dates of primary gas standards will be the expiration date assigned by the vendor. If the vendor does not provide an expiration date, then the expiration date is set to the date of receipt plus two years. Standards should be replaced sooner than the assigned date if there is evidence of degradation of the standard. The gases are stored at room temperature.

7.3.2 Calibration Standards: The primary 7 gas standard is made by Matheson Tri-Gas as a special custom order. The stock calibration standard mix is composed of nominally 1% (mole fraction) methane, ethane, ethene, acetylene, propane, isobutylene, and butane in nitrogen. A secondary 99% methane stock is obtained from Scott specialty gases and is used for the higher level methane standards (levels 3, 5, and 6). Pentane (Chromasolv, HPLC grade) from Sigma-Aldrich is added via a dilute solution in acetone. This solution is prepared by adding 4ul of neat pentane to 25 mL of acetone. The acetone is a high purity Ultra Resi Analyzed grade (99.4%) from Baker (other acetone solvents may be substituted as long as they do not contain interferences with the compounds analyzed with this method). This results in a stock concentration of 100ug/mL.

7.3.2.1 The initial calibration (ICAL) standards are prepared fresh each time an ICAL is performed.

7.3.2.2 To prepare the ICAL standards, a gas-tight syringe is used to transfer the following volumes of the stock standard to 22 mL vials that contain 18 mL of deionized water and 4 mL of headspace:

ICAL Level	Vol of Stock Calib Mix (uL)	Vol Stock Methane (uL)	Vol of Pentane Reagent (uL)
1	2.5	0	0.5
2	5	0	1.0
3	40	400	5.50
4	200	0	25
5	400	200	50
6	800	50	100
7	1000	0	125

7.3.2.3 The resulting seven ICAL levels are shown in Table 2. An example calculation explaining the derivation of these concentrations is shown in Attachment 1.

7.3.3 Initial Calibration Verification (ICV) Standard:

A second lot of gas mix prepared by Matheson Tri-Gas Inc. at a 1% concentration for each compound is used to prepare the ICV. The standard is prepared by adding 400 uL of the gas standard mixture to 18 mL of water. A second source of pentane, Chem Services cat# f2414s (conc 1000ug/mL), is used to prepare the ICV stock. The ICV stock is prepared at a concentration of 100ug/mL by diluting 2.5 mL of the vendor stock to 25 mL final volume. The ICV standard for analysis is then prepared by adding 50ul of the pentane ICV stock in addition to the 400ul

of gas to the 18 mL of water. Alternatively, a second source of the gas mix from Supelco, Scotty cat#23462 with just the methane, ethane, ethene and acetylene may be used for the ICV when only those compounds are being targeted for analysis. The concentrations of the gases in the ICV are as follows:

Concentration of Gases in ICV Standard

Gas	Concentration (µg/L)
Methane	146.0
Ethane	255.3
Ethene	273.7
Acetylene	237.0
Propane	401.4
Isobutylene	510.7
Butane	529.0
Pentane	278.2

7.3.4 Non-Routine Compounds

Other, non-routine compounds not listed in this section may be requested by a client and may be added to this procedure.

7.3.4.1 In these cases, all stock solutions will be obtained from commercial sources and will be verified with a second-source standard as described in Section 7.2 above.

7.3.4.2 Non-routine standards will be stored and treated as described in Section 7.3.1 above or as specified by the manufacturer.

7.3.4.3 Subsequent dilutions of specially requested compounds will be determined in a manner consistent with the client's recommendations for number of calibration points, inclusion of reporting limit, and concentration range adequate to represent the linearity of the instrument.

7.3.4.4 These specially requested, non-routine compounds either may be added to the dilution scheme used for routine compounds or may be prepared as a separate calibration.

7.3.4.5 All standards preparation for non-routine compounds shall be documented using the same method that is used for routine compounds.

7.3.5 Continuing Calibration Verification (CCV) Standard:

CCV standards are prepared from the same Matheson Tri-Gas gas stock as the ICAL standards. The concentrations of the gases in the CCV are

equivalent to the Level 4 standards shown in Table 2.

7.3.6 Laboratory Control Sample:

The LCS is fortified with the second source gas mix at the concentration of two times the level 4 calibration standard (see Table 2) using deionized water via cannula transfer in the same fashion as the samples.

7.3.7 Matrix Spike/Matrix Spike Duplicate:

The matrix spike (MS) and matrix spike duplicate (MSD) is created by fortifying a portion of an actual sample with the second source gas mix at two times the concentration of the level 4 calibration standard (see Table 2) in the same fashion as the calibration standards.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 The AFCEE QAPP, both version 3.1 and 4.0, do not require preservation with HCl and allow a holding time of 14 days. For non-AFCEE work, refer to the table below.

NOTE: Effervescing of carbon dioxide from alkaline waters will cause loss of the compounds of interest. Samples should be collected without acid if effervescence is observed. Exceptions such as this should be noted on the COC at the time of collection.

8.2 Care should be taken that no headspace is present in the sealed vials.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	VOA vial	40 mL	HCL, pH < 2; Cool, <6°C Not frozen	14 Days	Source Method (See Section 16.1)

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are

described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Method Performance Studies

An initial demonstration of capability (IDOC), retention time study, and a method detection limit study must be performed before using this method for the first time or if significant changes are made to the method. Afterward each new analyst must complete and IDOC and on-going proficiency must be demonstrated by each analyst on an annual basis. Current MDLs are found in the laboratory LIMS system. See Section 13 of this SOP for further details.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process. See QC Policy DV-QA-003P for further details.

9.4 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of 18 mL of deionized water transferred via cannula. The method blank is subject to the entire extraction and analysis process. The acetone that is used to prepare the pentane stock solutions should be lot tested by preparing a solvent check. The solvent check is prepared by adding 125ul of the acetone to 18 mL of the water that is used for the MB and is analyzed to the same specifications as listed here for the MB.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above ½ the reporting limit or above one-tenth of the concentration found in the associated samples. For DoD QSM 4.2 or 5.0 the acceptance criteria is no analytes

detected > ½ the RL (i.e., LOQ) or >1/10 the regulatory limit whichever is greater.

Corrective Action: If the method blank exceeds allowable levels, the source of the contamination should be investigated and all associated samples re-extracted and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample (LCS)

An LCS is prepared and analyzed with each batch of samples. The LCS is prepared as described in Section 7.3.6.

Acceptance Criteria: Control limits are 75-125%. In-house limits may be applied as described in Policy DV-QA-003P but must be no wider than 75-125%.

Corrective Action: If LCS recoveries are outside of the established control limits, and the MS/MSD recoveries are also out of control limits then the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike Sample (MS) and Matrix Spike Duplicate (MSD)

One MS/MSD pair is required with each analytical batch. The MS and MSD samples are prepared as described in Section 7.3.7. For the DoD QSM 4.2 or QSM 5.0, the MS/MSD must be from the project site and if insufficient sample is available to analyze the MS/MSD pair, this is documented in an NCM but no LCSD is performed. Likewise the analyst should inform the project manager, through an NCM, if the MS/MSD is not specified by the client but is prepared using another sample from another client's project.

Acceptance Criteria: The MS/MSD recoveries and relative percent difference (RPD) between the two results must be within historical control limits, which are based on three standard deviations of past results, using a minimum of 20 points. QC limits are reviewed on an on-going basis as QC data are developed, and limits are updated semi-annually. Current control limits are in the laboratory LIMS system. The relative percent difference must be no wider than ± 20%.

Corrective Actions: If MS/MSD recoveries or the RPD exceed acceptance limits, the entire batch control data must be evaluated. If analytical issues are suspected to be the cause for the out of control MS/MSD then fix any problems with the system and reanalyze the MS/MSD. If the preparation is suspected to be the cause of the out of control MS/MSD then the batch should be evaluated for re-preparation. If the MS/MSD is out of control and the LCS is in control, the data will be flagged as outside of control limits. This condition will be included in the final report case narrative. Otherwise, the affected samples should be reprepared and reanalyzed.

9.7 Sample Duplicate (DU)

One sample duplicate is required with every 10 samples.

Acceptance Criteria: The duplicate relative percent difference (RPD) between the two results must be ≤20%.

Corrective Actions: If the RPD exceeds acceptance limits, the samples must be reanalyzed. If the RPD exceeds upon reanalysis, report the data with the appropriate qualifier. In cases where the results are less than the RL and estimated this condition may be noted in an NCM and no further action is necessary.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and under the public folder, *Arizona Calibration Training.*

10.1.2 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Table 1 for typical operating conditions.

10.2 Initial Calibration (ICAL)

An initial calibration (ICAL) is performed after installing a new column and prior to analyzing samples. An ICAL is also performed whenever the initial or continuing calibration verification (ICV or CCV) analysis fails acceptance criteria, following major repair of the instrument, following maintenance that affects data quality, or when, in the judgment of the analyst, the GC performance is suspect.

10.2.1 The laboratory analyzes 7 calibration levels (as shown in Table 2) for the dissolved gases. The lowest point on the calibration curve is below the standard RL. The preparation of the calibration standards is described in Section 7.3.2.

10.2.2 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours. The ICV should be analyzed after the final calibration point is analyzed and accepted.

10.2.3 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.2.3.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

10.2.3.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.2.3.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.2.3.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average calibration factors or linear regressions, or six levels for second-order curve fits.

10.2.4 Calibrations are modeled either as average response factors (RF) or as calibration curves, using a systematic approach to selecting the optimum calibration function in order as described in the following sections.

10.2.5 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

10.3 External Standard Calibration

External standard calibration involves the comparison of instrument responses (e.g., peak area or peak height) from the target compounds in the sample to the responses of the target compounds in the calibration standards. The ratio of the detector response to the amount or concentration of target analyte in the calibration standard is defined as the calibration factor (CF), as follows:

$$CF = \frac{A_S}{C_S} \qquad \textit{Equation 3}$$

Where:

A_s = Peak area (or height) of the analyte or surrogate in the calibration standard.

C_s = Concentration of the analyte or surrogate, in ng/mL, in the injected calibration standard.

10.4 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until the calibration acceptance criteria are met.

10.4.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte in each calibration level against the concentration injected. For each analyte in each calibration standard, calculate the calibration factor (CF) as shown in Equation 3 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

$$AverageCalibrationFactor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \textit{Equation 4}$$

Where:

CF_i = Calibration factor for the ith calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\% \qquad \textit{Equation 5}$$

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \textit{Equation 6}$$

10.4.2 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor.

Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Note: The use of grand average (evaluation of the average response over all the compounds) is no longer allowed. Each compound must meet the RSD criteria.

Acceptance Criteria: The RSD must be ≤ 30%.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.4.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b \qquad \textit{Equation 7}$$

Where:

- y = Instrument response (peak area or height).
- x = Concentration of the target analyte in the calibration standard.
- a = Slope of the line.
- b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b \qquad \textit{Equation 8}$$

To calculate the concentration in an unknown sample extract, the regression equation (Equation 6) is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a} \qquad \textit{Equation 9}$$

Where:

A_s = Area of the chromatographic peak for the target analyte in the calibration standard.

A_e = Area of the chromatographic peak for the target analyte in the sample extract.

a = Slope of the line as determined by the least-squares regression.

C_s = Concentration of the target analyte in the calibration standard.

C_e = Concentration of the target analyte in the sample extract.

b = Intercept of the line as determined by the least-squares regression.

10.4.3.1 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. The August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the intercept must be significantly less than the reporting limit, and preferably less than the MDL. Also examine the residuals (as discussed above), but pay particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient ≥ 0.990. Some programs (e.g., AFCEE) require a correlation coefficient of ≥ 0.995. DoD QSM 5.0 requires the coefficient of determination to be >0.99.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted

10.4.4 Second-Order Regression

Calibrations using second-order regression produce a calibration function in the following form:

$$y = ax^2 + bx + c$$

Where:

y	=	instrument response for analyte (area)
x	=	concentration of standard (ug/L)
a	=	factor for second-order term (curvature)
b	=	factor for linear term
c	=	intercept

NOTE: Second order curves are not allowed for South Carolina work.

10.4.4.1 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria: The coefficient of determination must be ≥ 0.990.

Second-order regressions should be the last option. Note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- The distribution of concentrations is adequate to characterize the curvature.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the

problem must be investigated and the instrument recalibrated. Third-order regressions are not allowed at TestAmerica Denver.

10.5 Initial Calibration Verification (ICV)

Analysis of an ICV is performed after each ICAL. The preparation of the ICV standard is described in Section 7.3.3.

Acceptance Criteria: The result for the ICV must be within ± 20% of the expected value on both columns.

Corrective Action: If the measured value of the ICV is more than 20% different from the expected value, the accuracy of the standards used for calibration should be re-verified. If a problem is found then correct the issue and reanalyze the ICV. The instrument must be recalibrated before analyzing samples.

10.6 Continuing Calibration Verification (CCV)

A CCV is analyzed after every 10 samples and at the end of the analytical sequence. Some programs require that the concentration of the CCV be analyzed at two concentrations (a mid point and one at the RL) when second order calibration fits are used.

Acceptance Criteria: Any analyte that is reportable as found must be bracketed by acceptable CCVs on both columns. A CCV result is acceptable if the absolute value of the difference between the result and the accepted value is ≤20%.

In some cases, the nature of the samples being analyzed may be the cause of the failing %D. When the %D for an analyte falls outside of ± 20% in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed (at a dilution if column damage is eminent) to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client.

Corrective Action: If the acceptance limit is exceeded on either column, corrective action must be taken. This may include clipping the column or other minor instrument adjustments, followed by reanalyzing the standard. If the criterion is still not met, a new calibration curve must be prepared and all samples analyzed since the last successful CCV must be reanalyzed.

As an alternative DoD QSM 5.0 allows the laboratory to analyze two additional consecutive CCVs within one hour of the failed CCV. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and recalibrate: then reanalyze all affected samples since the last acceptable CCV.

If a DoD client accepts TestAmerica's Technical Specifications for DoD QSM work, samples that have no detections when a CCV has recoveries above the project acceptance limits would be reported with a case narrative comment, in addition to applying any data qualifier flags required by the project.

10.7 Retention Time Windows

Retention time (RT) windows must be determined for all analytes.

10.7.1 Determine new RT windows each time a new column is installed, after a major adjustment to the instrument conditions, or annually, whichever is most frequent.

10.7.2 Make an injection of all analytes of interest each day over a 72-hour period.

10.7.3 Calculate the mean and standard deviation for the three RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RT_i - \overline{RT}\right)^2}{n-1}}$$

Equations 12 & 13

Where:

RT_i = Retention time for the ith injection.
n = Number of injections (typically 3).
SD = Standard deviation.

10.7.3.1 Set the width of the RT window for each analyte at ± 3 standard deviations of the mean RT for that analyte.

10.7.3.2 The center of the RT window for an analyte is the RT for that analyte from the last of the three standards measured for the 72-hour study.

10.7.4 The center of the window for each analyte is updated with the RT from the level 4 standard of the ICAL or the CCV at the beginning of the analytical sequence. The width of each window remains the same until new windows are generated following the installation of a new column, or in response to an RT failure. The RT window width may be expanded if

the RT drift observed in the ICAL is greater than the established window. The expanded window is noted on the ICAL checklist.

10.7.5 If the RT window as calculated above is less than ± 0.03 minute, use ± 0.03 minute as the RT window. This allows for slight variations in retention times caused by sample matrix.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Transfer 18 mL of the sample using a 12” cannula needle to a tare weighed 22 mL serum vial with a crimp septum cap that is marked to contain approximately 18 mL. Insert a pressure relief valve (needle or cannula) through the septum about 3 mm into the 22 mL headspace vial. Insert a transfer cannula into the 40 mL VOA sample vial about ¾ of the way down into the vial. Insert the other end of the cannula into the 22 mL headspace vial all of the way to the bottom of the vial. Pressurize the 40 mL VOA vial with a stream of N2 gas with a flow of about 14-20 mL per minute. Remove the pressure gas after about 15 mL of volume has been transferred and pull the cannula out of the headspace vial when the 18 mL transfer is complete and allow the pressure to equilibrate through the pressure relief for a few seconds. Pull the cannula to above the liquid in the 40 mL VOA vial (to stop the flow of liquid). Remove the pressure relief valve. Reweigh the headspace vial to determine the volume transferred. Shake the headspace vial vigorously for 10-15 seconds and store inverted until ready to load on the headspace analyzer.

11.4 Measure the pH of the remaining sample using a pH strip and record it in the run log. Prepare an NCM for any samples with a pH > 2.

11.5 Instrument Troubleshooting and Maintenance

Before the start of any daily sequence the instrument should be evaluated for possible maintenance. Generally the instrumentation used for this method does not require any routine maintenance.

11.5.1 If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control.

11.5.2 A reduced response may indicate that the system needs to be evaluated for leaks.

11.5.3 Poor peak shape may be an indication that the columns need to be replaced.

11.5.4 The most common maintenance issues are typically related to a malfunctioning sample table and autosampling apparatus. If a malfunction is suspected (or expected) then service the apparatus to restore it to a normal operating condition.

11.5.5 Re-calibration should not be used to correct for maintenance related issues. Always document any maintenance procedure in the maintenance logbook.

11.6 Allow all samples and standards to warm to room temperature before injection. Samples are automatically injected onto two columns. Samples, standards, and QC samples must be introduced into the GC using the same procedure.

11.6.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data. Use the ChemStation chromatography data system to set up GC conditions for calibration. See Table 1 for typical operating conditions.

11.6.2 Prepare calibration standard solutions in headspace vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

11.6.3 Unprocessed calibration data are transferred to the Chrom database for processing. See Section 10 regarding selection of the appropriate calibration model. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

11.7 Load each sample, as well as the various standards and QC samples, into the autosampler.

11.8 The daily run sequence is documented in a bound log book, and should be as follows:

Primer (generally the primer is run twice)

Daily opening CCVs

LCS

LCSD

Method Blank

9 samples plus duplicate

CCVs

Followed by cycles of 9 samples, second duplicate, and CCVs as needed

Closing CCV

11.9 Daily Retention Time Windows

The center of the retention time (RT) windows determined in Section 10.7 are adjusted to the RT of each analyte as determined in the 12-hour calibration verification. The centers of the RT windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration, but not for any other calibration verification standards.

11.10 Upon completion of the analytical sequence, transfer the raw chromatography data to the Chrom database for further processing.

11.10.1 Review chromatograms online and determine whether manual data manipulations are necessary.

11.10.2 All manual integrations must be justified and documented. See DV-QA-011P, *Acceptable Manual Integration Requirements.*

11.10.3 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

11.10.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration. Manual integrations that are documented in this manner must be scanned into the documents section of the analytical batch.

11.11 Compile the raw data for all the samples and QC samples in a batch. The preparation batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.11.1 The data package should consist of the checklist, sequence(s), ICAL cover, ICAL summary and history used for data quantitation and the prep batch paperwork. The sequence(s) and ICAL summary should be scanned into the TALS database documents section.

11.11.2 Perform a level 1 data review and document the review on the data review checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.11.3 Submit the data package and review checklist to the peer reviewer for the level 2 review. All manual integrations must be evaluated by the peer reviewer and this review must be documented by date and initial on the manual integration summary report and/or the level 2 review checklist. For Federal projects and certain client specified projects, the documentation of the manual integration review must be scanned and

attached to the project in the LIMS to be included with the Level 4 data package. The level 2 review is documented on the review checklist initiated at the level 1 review. The data review process is explained in SOP DV-QA-0020.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

12.1.1 Tentative identification occurs when a peak is found on the primary column within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if qualified data (J flags) are to be reported. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmation column. Absolute retention times are adjusted according to the retention times of the daily opening CCV.

12.1.2 The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2 Corrective Action for Retention Times

12.2.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established by the initial calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

12.2.1.1 The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

12.2.1.2 No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

12.3 Dual-Column Quantitation

12.3.1 Each sample is analyzed on two different columns at the same time. The laboratory designates a primary column based on optimal separation of the compounds of interest and other desirable chromatographic characteristics. The result from the primary column is normally reported. If the continuing calibration verification fails on one of the columns, the appropriate corrective action must be taken. (See Section 10.6.) The result from the secondary (confirmatory) column may be reported if any of the following is true:

12.3.1.1 There is obvious chromatographic interference on the primary column.

12.3.1.2 The difference between the result for the primary column and the result for the secondary column is > 40% and chromatographic interference is evident on the primary column.

12.3.1.3 For DoD QSM 4.2 or QSM 5.0 work, calibration and QC criteria for the second column are the same as for the initial or primary column analysis.

12.3.2 Dual Column Results With > 40% RPD

12.3.2.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram.

12.3.2.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.3.2.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.3.2.4 The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

12.4 Calibration Range and Sample Dilutions

12.4.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for the analyte(s) that were found to be over the calibration range in the high sample, they must be reanalyzed to rule out carryover, unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when samples or blanks were analyzed after another sample with similar high compound recovery or when the detection in the sample with suspected carryover is much higher than the expected amount of carryover (i.e. the sample's concentration may be similar to or

higher than the concentration found in the previous sample). It may also be necessary to dilute samples because of matrix interferences.

12.4.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.4.3 Prepare dilutions using the same reagent water as is used to prepare the MB and LCS. Transfer 18 mL of the diluent water into a tare weighed headspace vial marked to contain 18 mL of solution. Using a gastight syringe, carefully remove exactly the amount of water that you plan to add with sample and reweigh the headspace vial. Record this weight in the run logbook. Using a cannula to transfer the sample targeted for dilution carefully fill the vial (and diluent) to the 18 mL mark with sample. Reweigh the vial again and record this weight in the run logbook. Use the weights recorded for water and sample to calculate the exact dilution. For transferring sample volumes that are less than 2 mL (greater than an approximately 10x dilution) it is recommended that the analyst use a gastight syringe to measure the sample volume. The gastight syringe should be filled using positive pressure in order to avoid degassing the sample.

12.4.4 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.4.5 Reporting Dilutions

Some programs (e.g., South Carolina and AFCEE) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.5 Interferences in Observed in Samples

Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.6 Calculations

12.6.1 Spike Recovery Calculation

LCS, MS, and MSD spike recoveries are calculated using the following equation:

$$\%\ Recovery = \frac{Concentration\,(or\ amount)\ found}{Concentration\,(or\ amount)\,spiked} \times 100$$

12.6.2 MS/MSD RPD Calculation

$$RPD = \frac{R_1 - R_2}{\frac{1}{2}\left(R_1 + R_2\right)}$$

Where:

R_1 = result for the MS

R_2 = result for the MSD

12.6.3 Concentration of Analyte in Sample

The concentration of the analyte in the sample is calculated as follows:

$$C_S = C_C \times DF$$

Where:

C_s	=	concentration of analyte in sample (µg/L)
C_C	=	on-column concentration (ng/mL)
DF	=	Dilution Factor, post extraction dilutions

12.7 All data are subject to two levels of review, which is documented on a checklist (see SOP DV-QA-0020). See also Section 11.11.

13.0 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.1 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.1.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.1.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.1.4 Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

13.2 Training Requirements

13.2.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Prevention

Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, “Waste Management and Pollution Prevention”, of the Environmental Health and Safety Manual, and DV-HS-001P, “Waste Management Program.”

15.1 The following waste streams are produce when this method is carried out:

- Methanol Vial Waste – Waste Stream A
- Liquid Methanol Waste – Waste Stream C
- Acidified Water – Waste Stream W
- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 RSK SOP-175 Revision 0, R.S. Kerr Environmental Research Laboratory; August 11, 1994.

16.2 Method 8000B, "Test Method for Evaluating Solid Waste", EPA SW-846, Update III; December, 1996.

16.3 AFCEE Quality Assurance Project Plan, Version 3.1; August 2001.

16.4 AFCEE Quality Assurance Project Plan, Version 4.0, February 2005

17.0 Deviations From the Source Method

The method measures the concentration of the gases in the headspace and then calculates the concentration of the gas dissolved in the original water sample based on its partitioning properties, as indicated by its Henry's Law constant. This has been modified for this SOP by calibrating against the concentration of dissolved gases in water standards instead of the concentration of gases in the sample headspace.

18.0 Attachments

Table 1 – GC Operating Conditions
Table 2 – Initial Calibration Levels
Table 3 – Standard Reporting Limits

Attachment 1 – Example Calculation of ICAL Concentrations
Attachment 2 – Example Chromatogram
Attachment 3 – Example Chromatogram

19.0 Revision History

- Revision 6, dated 30 September 2014
  - Revised section 4.1 regarding carryover
  - Added information regarding location of electronic spreadsheet for pipette calibration in section 6.1.2
  - Added specification of instrument ID in section 6.2.1
  - Revised section 9.1 to reflect current practice
  - Added DoD QSM 5 QC and calibration criteria throughout
  - Added statement to Section 10.5 regarding corrective action for out of control ICV
  - Added information regarding frequency of determining RT window
  - Added new section 11.4, Instrument Troubleshooting and Maintenance
  - Added statement in Section 11.9.4 regarding documentation for manual integration processed manually.
- Revision 5, dated 31 July 2013
  - Added vial calibration information to section 6.1.2
  - Updated sections 7.3.2, 7.3.3, 9.4, 9.6, 9.7, 11.4.2, 11.9.1 and 12.4 to reflect

current practices
- Updated references to “Target” to “Chrom” through out document
- Added Chrom example chromatogram (Attachment 2)
- Added elution order and analyte number (table 3)

- Revision 4, dated 30 July 2012
  - Added references to new analytes throughout the SOP
  - Added reference to the fracking industry to Section 1.2
  - Updated Section 6 with new equipment/column information
  - Revised calibration standards to include new analytes and current concentrations
  - Expanded calibration section to include new analytes
  - Updated section 7.3.5 to current gas vendor.
  - Updated section 11.2 to reflect current practices
  - Updated tables to include new analytes
  - Added South Carolina requirements to Section 13.1

- Revision 3, dated 22 July 2011
  - Revised calibration standards
  - Expanded calibration section
  - Clarified requirement for calibration verifications to meet criteria on both columns, not just the primary column or column used for quantitation.

- Revision 2.3, dated 8 October 2010
  - Added items to supplies in section 6.1
  - Section 10.2 modified to utilize sample transferring process that eliminates exposure of the sample to the air during transfer to the headspace vials that are used by the instrumentation.
- Revision 2.2, dated 11 June 2010
  - Annual Technical review
  - Made grammatical and formatting corrections
  - Deleted the 4th dot in Section 5.1 (Purge & Trap not used)
  - Fixed Section 9.2.1.2 to be 30% RSD.

- Previous Changes made.
  - Major changes and updates were made, which resulted in significant changes in numbering compared to revision 0.
  - In section 2.2, deleted statement that calibration is performed daily. The frequency of the initial calibration is clarified in section 10.2.1.
  - Revised section 2.3 for clarity.
  - Rewrote section 5, Safety, to comply with current TestAmerica requirements.

- Incorporated Interim Change A into section 7.2.
- In section 7.2.1.2, added 2 more levels to the calibration.
- In section 7.2.1.3, added table with concentration values.
- In section 7.2.3, corrected the text to state that the matrix spike is made using a portion of an actual sample, instead of reagent water.
- Added section 10.2.1 to explain when initial calibrations are performed.
- Added section 10.2.9 to provide calculation for second order regression.
- Revised section 10.4.2 for clarity.
- Added section 11.2 to provide step for measuring and recording pH of samples.
- Added section 11.3 to provide step for transferring 18 mL of sample to the serum vial.
- Added section 11.5 to provide step for loading autosampler.
- In section 12.2.3, added explanation of variables.
- In section 12.5.2, added explanation of variables.
- In section 12.5.3, clarified variables used.
- Added section 17, which was missing from revision 0.
- In Appendix 1, corrected calculations.
- Added Table 1 to provide GC operating conditions.
    - Modifications made for this “dot” revision:
    - Corrected vial specification in Section 8.1 from “43 mL vials” to “three 40 mL vials.”
    - Revision 2 – February 1008
    - Changed to TestAmerica new format and changed name to TestAmerica Denver

TABLE 1 – GC Operating Conditions

Parameter	Recommended Conditions
Injection Port Temperature	200° C
Detector Temperature	250° C
Oven Temperature	45°C held for 2.4 minutes, ramped at 19.33°/min to 74°, ramped at 1°/min to 77° and held for 0.25 minutes, ramped at 30°/min to 125° and held for 2.6 minutes.
Column 1	RT-U-Bond 30 m x 0.32 mm x 10 um
Column 2	RT-S-Bond 30 m x 0.32 mm x 10 um
Injection Volume	1 mL
Carrier Gas and Flow Rate	Helium 30 psi (~8.2 mL/min) held for 2.5 minutes, ramped at 10psi/min to 40psi and held for 1 minute (~11.3mL/min), ramped at 15psi/min to 55psi (~18.5mL/min, decreasing to ~15.2mL/min at 125°)
Detector Gases and Flow Rates	Hydrogen/Air 40 mL/min, makeup nitrogen at 25 mL/min
Y Splitter	Yes

TABLE 2 – Initial Calibration Levels

	Concentration in Aqueous Phase (µg/L)[1]						
Compound	**Level 7**	**Level 6**	**Level 5**	**Level 4**	**Level 3**	**Level 2**	**Level 1**
Methane	365.0	2098	7373	73.00	14470	1.825	0.8125
Ethene	638.3	510.6	255.3	127.6	25.53	3.191	1.595
Ethane	684.2	547.4	273.7	136.8	27.37	3.421	1.710
Acetylene	592.5	474.0	237.0	118.5	23.70	2.962	1.481
Propane	1003	802.8	401.4	200.7	40.14	5.017	2.508
Isobutylene	1276	1021	510.7	255.3	51.07	6.384	3.192
Butane	1322	1058	529.0	264.5	52.90	6.613	3.306
Pentane	695.5	556.4	278.2	139.1	27.82	5.564	2.782

[1]These are estimated concentrations. Actual concentrations will vary slightly when the calibration standards are prepared.

TABLE 3 – Standard Reporting Limits

Compound	(ug/L)
Methane	5.0
Ethane	5.0
Ethene	5.0
Acetylene	5.0
Propane	5.0
Isobutylene	5.0
Butane	5.0
Pentane	5.0

TABLE 4 – Elution Order and Analyte Number

Analyte	Column A	Column B
Methane	**1**	**1**
Ethylene	**2**	**2**
Ethane	**3**	**NA**
Acetylene	**5**	**NA**
Acetylene/Ethane	**NA**	**4**
Propane	**6**	**6**
ButaneIsobutylene	**8**	**NA**
Butane	**NA**	**9**
Pentane	**10**	**10**

Attachment 1 – Example Calculation of ICAL Concentrations

This example is for methane at the 7th calibration level, where 1,000 µL of the stock standard (10,000 ppmV) is used.**.**

The calculation is based on the Ideal Gas Law: PV = nRT

Where:

P	=	pressure in atmospheres (760 mm Hg = 1 atmosphere)
V	=	volume of gas in liters
n	=	number of moles of gas
R	=	gas constant, i.e., 08206 atm L / mol K
T	=	temperature in degrees Kelvin (T = °C +273.15)

Calculations for the standards are based on 22°C at 754 mm Hg, therefore

P	=	754/760 = 0.99211 atm
V	=	1 L of stock standard gas
R	=	0.08206
T	=	22 + 273.15 = 295.15 °K

Solving for n,

$$n = \frac{PV}{RT} = \frac{0.99211 atm \times 1L}{0.08206 atm \bullet L / mole \bullet K \times 296.15K} = 0.04082 \text{ moles of methane in 1 L standard}$$

NOTE: Daily variations in laboratory temperature and pressure create less than 1% variation.

The following equation is used to calculate the concentration of methane in the Level 7 ICAL standard:

$$\frac{0.001\,L\,\mathrm{N_2}}{0.018\,L\,\mathrm{H_2O}} \times \frac{0.01\,L\,\mathrm{CH_4}}{1.0\,L\,\mathrm{N_2}} \times \frac{0.04082\,mole\,\mathrm{CH_4}}{1.0\,L\,\mathrm{CH_4}} \times \frac{16.04\,g\,\mathrm{CH_4}}{1.0\,mole\,\mathrm{CH_4}} = 0.00036375\,g\,\mathrm{CH_4}/L\,\mathrm{H_2O} =$$

363.8 *µg/L* methane concentration in Level 7 ICAL standard

Where:

0.001 L N_2	=	Volume of gas (in L) used to prepare the aqueous standard.
0.018 L H_2O	=	Volume of water in which the standards are prepared.
0.01 L CH_4	=	Volume of methane in 1.0 L of the gas standard, which is a 1% methane mixture in N_2
0.04082 mole CH_4	=	Number of moles of methane in 1.0 L of methane as determined above
16.04 g CH_4	=	Molecular weight of methane

Attachment 2
Example Chromatogram

TestAmerica Denver

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Title: Acid-Soluble and Acid-Insoluble Sulfides: Distillation and Titration [SW 9030B/ SW9034]

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1.0 Scope and Application

1.1 This SOP covers both the sample pretreatment procedure designed to remove interfering substances prior to analysis and the determination of the concentration of acid-soluble sulfide. It is based on SW-846 Methods 9030B and 9034.

1.2 This method does not measure acid-insoluble sulfides.

1.3 This method is applicable to the measurement of total acid-soluble sulfides in drinking water, surface and saline waters, domestic and industrial wastes and solid waste materials.

1.4 The reporting limit for water samples is 4 mg/L and for soil samples is 5 mg/Kg. Samples that contain more than 50 ppm sulfide should be diluted.

2.0 Summary of Method

2.1 The sulfide method is a two part method involving the distillation of the sample followed by titration of the collected scrubber solution.

2.2 The separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample in a closed system. The sample is then heated (for total sulfide) to 70 ℃. The H_2S, which is formed, is carried by a nitrogen stream into a zinc acetate/formaldehyde gas scrubbing bottle. Under these conditions, it is precipitated out as ZnS.

2.3 The titration is accomplished by the addition of excess iodine to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodide oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

3.0 Definitions

Please refer to the Glossary of the TestAmerica Denver Quality Assurance Manual and SOP DV-QA-003P for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Aqueous samples should be taken with a minimum of aeration to avoid the oxidation or volatilization of sulfide.

4.2 Reduced sulfur compounds, such as sulfite and bisulfite decompose in acid to form SO_2. The SO_2, if carried over into the scrubbing solution may yield false positives. The addition of formaldehyde in the scrubber solution removes this interference. This method shows no sensitivity to sulfite or bisulfite at concentrations up to 10 ppm.

4.3 Many metals (e.g., Hg, Cd, Cu) form insoluble sulfides and give low recoveries.

4.4 The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds, both

solid and dissolved. These compounds are common in some sample types, such as refinery waste and boiler feed water. Where these compounds are known to be present, the zinc hydroxide/sulfide precipitate can be centrifuged and washed with deionized water to remove the interferent.

4.5 High levels of color and turbidity may interfere.

4.6 If water samples are not preserved with zinc acetate, analysis must start immediately.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.3.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3.4 Hydrogen sulfide (H_2S) gas is generated by the addition of sulfuric acid. Inhalation of H_2S gas can cause headache, dizziness, nausea, and unconsciousness and potentially death.

5.3.5 Acid should be added dropwise until the sample reactivity is observed, particularly for soil samples and discolored water samples. If a strong reaction is observed, increase stirring rate and continue to add slowly.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section.

Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Formaldehyde	Toxic	0.75 ppm (TWA)	Inhalation may result in spasm, inflammation, and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Iodine	Poison Corrosive Oxidizer	0.1 ppm-Ceiling	Vapors severely irritate and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Sulfide	Corrosive	10 ppm (TWA) 15 ppm (STEL)	Will form hydrogen sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
Sodium Hydroxide	Corrosive	2 mg/m^3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Nitrogen gas.

6.2 Reaction vessels, 500 mL round-bottom flasks with 3 necks, 24/40 joints.

6.3 Addition funnels, 125 mL, 24/40 outlet joint.

6.4 Scrubber bottle, 250 mL.

6.5 Tygon tubing.

6.6 Glass tubing.

6.7 Hotplate, capable of maintaining 70℃ ± 5℃.

6.8 5 mL autopipettor

6.9 Burette, 25 mL, Class A.

6.10 Erlenmeyer Flasks, 250 mL and 500 mL.

6.11 Magnetic stirrer plate, magnetic stirrer bars, optional.

6.12 Assorted analytical glassware, as needed.

6.13 pH strips

6.14 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 All reagents listed herein shall be ACS Reagent Grade, unless specified otherwise.

7.2 Reagent Water

Free of substances that interfere with analytical methods. Water with a resistivity of 1 megohm-cm or greater. The TestAmerica Denver house deionized water meets this requirement with a minimum resistivity of 10 megohm-cm.

7.3 Reagent sand, such as Ottawa sand for use as blank solid matrix.

7.4 Sulfuric acid, concentrated. (H_2SO_4).

7.5 Hydrochloric acid, concentrated (HCl).

7.6 Sodium Sulfide nonahydrate crystals (SFD ICV STK or SFD CAL STK)

7.7 Sulfide Standard Solution (Na_2S), approximately 1000 mg/L (SFD CAL INT or SFD ICV INT)

Dissolve approximately 3.75 g of $Na_2S \bullet 9H_2O$ (Section 7.5) crystals in 400 mL reagent water in a 500 mL volumetric flask. Add 2 mL of 50 % sodium hydroxide and dilute to 500 mL. Standardize daily according to Section 10.4.1

7.8 Zinc acetate dihydrate (ZINC ACETATE)

7.9 Zinc acetate, 0.5 M (znac)

Dissolve 110 g of zinc acetate dihydrate (Section 7.7) in 200 mL of deionized water. Add 1 mL of concentrated HCl and dilute to 1 liter.

7.10 Formaldehyde, 37% (form)

Obtain solution from a commercial vendor.

7.11 Hydrochloric Acid, 6 N (1:1) (HCL Sol)

Very carefully and slowly, and with constant mixing, add 250 mL of concentrated hydrochloric acid (HCl) to 250 mL of reagent water and mix. Allow to cool before use.

7.12 Starch Indicator (Starch Ind)

7.12.1 The starch indicator solution is purchased commercially.

7.12.2 Alternatively, to prepare the indicator solution in the lab, dissolve 2 g of laboratory-grade soluble starch and 2.0 g of salicylic acid, as a preservative, in 100 mL of hot reagent water. Mix, cool, and store in a labeled poly bottle.

7.13 Sodium Thiosulfate, 0.0250 N (Na thio)

This solution is purchased as a standardized 0.0250 N solution.

7.14 Iodine Solution, 0.0250 N (Iod)

This solution is purchased as a standardized 0.0250 N solution.

7.15 Sodium Hydroxide (NaOH), 50%

7.15.1 This solution is usually purchased from commercial sources.

7.15.2 If prepared in the lab, very carefully and slowly, with constant stirring, add 250 g of NaOH to 200 mL of reagent water in a beaker.

CAUTION: This solution gets very hot. Add the NaOH to the water slowly to allow the heat to dissipate.

7.15.3 Allow to cool, then transfer the solution to a 500 mL volumetric flask and dilute to volume with reagent water.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE	250 mL	NaOH/Zn Acetate pH>9 Cool, $\leq 6^{o}C$	7 Days	SW-846
Soil	Glass	4 oz jar	Cool, $\leq 6^{o}C$	7 Days	SW-846

NOTE: Samples should be collected with a minimum of aeration. **The sample bottle should be filled completely, excluding all head space, and sealed.** Analysis should commence as soon as possible, and samples should be kept in a cool, dark place until analysis begins.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via method comments in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends.

The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Sample QC - The following quality control samples are prepared with each batch of samples.

9.2.1 Method Blank

A minimum of one method blank must be included in each QC batch of 20 or fewer field samples. The method blank consists of reagent water for batches of aqueous samples or Ottawa sand for batches of solid samples. Prepare and analyze the blank in the same manner as samples.

Acceptance criteria: The result for the method blank must be less than one-half the reporting limit for the analyte of interest or less than 10% of the lowest analyte concentration found in the associated samples, whichever is higher. In the latter case, an NCM must be generated explaining the analyte detection in the blank. Note that some programs (e.g., DoD) require the maximum blank concentration to be less than one-half of the reporting or less than 10% of the lowest sample concentration.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed after first checking all reagents and glassware for sources of contamination, checking the condition and cleanliness of the burette delivering the titrant, and correcting any problems found.

If the analyte was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

If there is insufficient sample for reanalysis, an NCM must be prepared and the client contacted by the laboratory Project Manager.

9.2.2 Laboratory Control Sample (LCS)

A minimum of one LCS must be included in each QC batch of 20 or fewer field samples. The LCS is prepared by spiking 250 mL reagent water or 50 g of Ottawa sand with 5 mL of the Sulfide Standard Solution (Section 7.6). The nominal spike concentration for the water LCS is 20 mg/L and for the solid LCS is 100 mg/Kg. The LCS is analyzed in the same

manner as the sample distillates. A duplicate LCS (LCSD) may be prepared and analyzed to provide a measure of analytical precision if required by the client or project and in cases where there is insufficient sample to prepare an MS and MSD.

Acceptance criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean and must be no wider than the limits specified in the reference methods or by specific projects.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed. If reanalysis is not possible, an NCM must be prepared and the client contacted by the laboratory Project Manager.

9.2.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair is required with each batch of 20 or fewer samples for total sulfide. The MS and MSD are prepared by adding 5 mL of the Sulfide Standard Solution to the sample aliquot required (Section 7.6). If insufficient sample volume is available for the preparation of an MS/MSD or a sample duplicate, a duplicate LCS may be analyzed.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference (RPD) between the MS and MSD, or between the sample and sample duplicate, must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

10.3.1 Prepare the scrubber bottles by adding 235 mL of deionized water to a 250 mL bottle. Add 10 mL of zinc acetate solution and 5 mL of 37% formaldehyde.

10.3.2 Prepare reaction vessels, as follows:

10.3.2.1 Solids and Sludges

Weigh 50 g of soil or sludge into a 3-necked 500 mL round bottom flask. Add 250 mL of DI water and stopper immediately. The sample weight can be reduced to 25 g if the matrix absorbs water or does not stir efficiently.

10.3.2.2 Liquids

Check the pH of the sample with wide-range pH paper and record on the benchsheet. Shake the sample container to suspend any solids and then quickly transfer 250 mL of sample with a graduated cylinder into a 3-necked 500 mL round bottom flask and stopper immediately.

10.3.3 Assemble the apparatus as shown in Attachment 1. Add 25 mL of concentrated sulfuric acid to an addition funnel and connect it to the reaction flask. Connect the nitrogen stream to the flask as shown in Attachment 1. Be sure all fittings and joints are air-tight.

10.3.4 Adjust the nitrogen flow to about 5 bubbles per second in the scrubber bottle.

10.3.5 Once the gas flow has stabilized, purge the system for 15 minutes.

10.3.6 Add sulfide standard solution by pipette to the LCS and MS/MSD (if MS/SD are analyzed). Keep the tip of the pipette beneath the water

surface and seal the vessel up tightly after addition. Spiking is normally done with 5 mL of sulfide standard solution (Section 7.6).

10.3.7 Add water to the water bath on each hot plate and turn on each stirrer so that the sample is stirred gently, without vortexing.

10.3.8 Open the addition funnel so that the concentrated sulfuric acid drips slowly into the sample. Some samples may react violently with the acid, in which case the addition rate of acid may be decreased and the stir rate increased.

10.3.9 Once all acid has been added, turn on the hot plate so reaction vessels will heat to 70 ± 5 ℃.

10.3.10 Allow the reaction to proceed for 90 minutes, checking the temperature frequently. Record the start and end times of the distillation in the batch record.

10.3.11 Once the distillation is complete, use pH paper to check the pH of the spent solution in the reaction vessel. If the pH is not less than or equal to 1, reprepare the sample using more acid.

10.3.12 If the pH is less than or equal to 1, close the scrubber bottles and proceed to the analysis outlined in Section 10.5 for total sulfide determination by titration by Method 9034.

10.4 Calibration

10.4.1 Standardization of Sulfide Standard Solution

10.4.1.1 Pipette 20.0 mL of iodine solution (Section 7.14) into a 250 mL Erlenmeyer flask.

10.4.1.2 Add 2 mL of 6 N hydrochloric acid (Section 7.11).

10.4.1.3 Pipette 5.0 mL of the Sulfide Standard Solution (Section 7.6) into the flask, making sure that the delivery tip of the pipette is below the surface of the solution. Dilute to approximately 100 mL with reagent water.

10.4.1.4 Titrate with 0.0250 N sodium thiosulfate solution (Section 7.13) to a pale yellow straw color.

10.4.1.5 Add 1 mL of starch indicator solution (Section 7.12) and swirl until a homogenous blue color develops.

10.4.1.6 Continue the titration until the blue color disappears. Record the volume of the sodium thiosulfate titrant used.

10.4.1.7 Calculate the sulfide concentration using the sulfide titration spreadsheet (Attachment 3).

10.4.1.8 Perform the standardization titrations in duplicate. Use the average of the two results as the final sulfide concentration of the Sulfide Standard Solution.

10.5 Sample Analysis

10.5.1 Determination of Total Sulfide (Acid-Soluble Sulfides)

10.5.1.1 Transfer the contents of the bottle from 10.3.11 to a 500 mL Erlenmeyer flask. Gently swirl the collection bottle so that all precipitated zinc sulfide is collected. Rinse the bottle with reagent water to remove any remaining white precipitate and add the rinsate to the flask. The transfer should be accomplished with a minimum of agitation to minimize the exposure of the sample to air.

10.5.1.2 Add 2 mL of 6 N HCl (Section 7.11) to the distilled sample in the Erlenmeyer flask and swirl.

10.5.1.3 Using a automated pipette, dispense a known amount of 0.025 N iodine solution (Section 7.14) into the flask. The amount added needs to be in excess of the amount needed to oxidize the sulfide. This is typically 5 mL for "ND" samples and 20 mL for samples with sulfide in the range of the LCS samples. Add enough iodine solution to turn the liquid in the flask a deep amber color. Record the volume of iodine used.

NOTE: There must be enough iodine to oxidize the entire amount of sulfide present as zinc sulfide precipitate in the scrubber solution. Use about 1.5 mL of the 0.0250 N iodine solution for every mg of sulfide estimated to be in the sample. The amount of iodine necessary can be estimated by the amount of precipitate present in the scrubber solution. After addition of the iodine, the solution should be a deep amber color. If, in the analyst's opinion, the amount of iodine needed to oxidize the sulfide present in the sample will take more than 25 mL, a smaller sample aliquot may be taken for analysis. This volume must be measured and recorded.

10.5.1.4 If desired, add a stir bar and place the flask on a magnetic stirrer.

10.5.1.5 Titrate the solution with 0.0250 N sodium thiosulfate solution (Section 7.13) to a pale yellow color.

10.5.1.6 Add sufficient starch indicator solution (Section 7.12) to achieve a deep and homogeneous blue color. Typically this volume is approximately 1 mL.

10.5.1.7 Continue the titration until the blue color just disappears. Allow the sample to equilibrate to ensure the endpoint is not missed.

10.5.1.8 Record the volume of sodium thiosulfate solution used.

10.6 Troubleshooting and Maintenance

10.6.1 Ensure the Nitrogen inlet tube is below the sample surface and is not clogged.

10.6.2 Check the ground glass joints for light grease and not excessive grease.

10.6.3 Check the tubing for cracks or signs of frailty or excessive wear.

10.6.4 Ensure the inlet and out tubes for the nitrogen are not chipped.

11.0 Calculations / Data Reduction

11.1 All calculations are performed in the sulfide titration benchsheet (spreadsheet). For manual verification use the following calculations.

11.2 One mL of 0.0250 N iodine solution reacts with 0.4 mg of sulfide present in the titration flask. Use the following equation to calculate sulfide concentration:

$$S = \frac{[(A \times B) - (C \times D)] \times 16000}{V_s}$$ *Equation 1*

Where:

S = Concentration of sulfide in sample (mg/kg or mg/L)
A = Volume of iodine solution added (mL)
B = Normality of iodine solution
C = Volume of sodium thiosulfate solution (mL)
D = Normality of sodium thiosulfate solution
V_s = Volume (mL) or weight (g) of original sample aliquot

11.3 If the normality of the iodine solution is exactly the same as that of the sodium thiosulfate solution, i.e., 0.0250 N, the following calculation may be used instead of the equation in section 11.2:

$$S = \frac{(A - C) \times 400}{V_s}$$ *Equation 2*

Where:

S = Concentration of sulfide in sample (mg/kg or mg/L)
A = Volume of iodine solution added (mL)
C = Volume of sodium thiosulfate solution (mL)
Vs = Volume (mL) or weight (g) of original sample aliquot

11.4 LCS Percent Recovery

Use the following equation to calculate the percent recovery of sulfide in the LCS:

$$\text{LCS } \%\text{Recovery} = \frac{\text{Measured Value}}{\text{True Value}} \times 100\% \qquad \textit{Equation 3}$$

11.5 MS and MSD Percent Recovery

Use the following equation to calculate the percent recovery of the added sulfide in the MS and MSD samples:

$$\text{MS } \%\text{Recovery} = \frac{SSR - SR}{SA} \qquad \textit{Equation 4}$$

Where:

SSR = Spiked sample result (concentration of sulfide in the MS or MSD).

SR = Sample result (concentration of sulfide in the parent sample).

SA = Concentration of added spike (concentration of sulfide in the sample in addition to any native sulfide as the result of adding the spiking solution).

11.6 Relative Percent Difference (RPD)

Use the following equation to calculate the relative percent difference between two analytical results (e.g., MS and MSD, LCS and LCSD, sample and sample duplicate).

$$RPD = \frac{|(R_1 - R_2)|}{1/2\,(R_1 + R_2)} \times 100\% \qquad \textit{Equation 5}$$

Where:

R_1 = Result for first sample (MS, LCS, or sample).
R_2 = Result for duplicate sample (MSD, LCSD, or sample duplicate).

11.7 All data undergo first and second level review using a checklist. See SOP DV-QA-0020 for a copy of the checklist and for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL Policy DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.1.2 There is no true MDL for this method. For the purposes of method detection the smallest increment of titrated volume is calculated and used as the MDL. Whenever a new buret is used the volume is rechecked and put through the calculation.

12.1.3 The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.2 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.3 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.4 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Scrubber waste – Aqueous Acidic (Reactivity) - Waste Stream F

14.2.3 Contents of reaction vessel – Aqueous Acidic (Reactivity) - Waste Stream F

14.2.4 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.1.1 Method 9030B: Acid-Soluble and Acid-Insoluble Sulfides: Distillation, Revision 2, December 1996.

15.1.2 Method 9034, Titrimetric Procedure for Acid-Soluble and Acid Insoluble Sulfides, Revision 0, December, 1996.

17.0 Method Modifications:

Item	Method	Modification
1	SW 9030B	In this SOP the distillation apparatus uses only one scrubber.
2	SW 9030B	For total sulfide, 25 mL of concentrated sulfuric acid is added to the system without first titrating the sample. After the 90 minute reaction period, the spent sample solution pH is measured. If the pH is not ≤ 1.0, the sample is re-distilled using additional sulfuric acid.

Item	Method	Modification
3	SW 9034	Rinse solution of standardized 0.0250 N iodine, 1 mL of 6 N HCl, and reagent water is not used in the above procedure. As samples are preserved with 1% sodium hydroxide and zinc acetate, and the total sulfides are preserved with formaldehyde and zinc acetate, rinsing the bottles with reagent water introduces very minimal loss of sulfide due to oxidation.
4	SW 9034	The sodium thiosulfate and iodine solutions are purchased standardized from a vendor and therefore are not standardized in the laboratory.
5	SW 9034	The Method 9034 procedure is to add the iodine solution into a 500 mL flask, bring to 100 mL with reagent water, add acid, and pipette the scrubber solutions from method 9030 under the iodine solution. The scrubber solutions are approximately 200 mL. Due to the large volume of scrubber solution, the laboratory transfers the scrubber solutions into a flask and then adds the acid and iodine solution.
6	SW 9030B	Soil samples are refrigerated upon receipt and are not preserved with zinc acetate. The laboratory uses the 7 day holding time stated in SW-846. Since sample composition and moisture content vary so widely, it is difficult to preserve the sample in the field as prescribed. It is recommended that the sample be taken with no headspace.
7	SW 9030B	Aqueous samples are measured volumetrically in graduated cylinders, not by weight as indicated in the source method. Generally the liquid samples received by the lab are water rather than non-aqueous wastes.

18.0 Attachments

Attachment 1: Distillation Apparatus
Attachment 2: Example Sample Preparation Benchsheet - LIMS
Attachment 3: Example Titration Benchsheet – Excel Spreadsheet
Attachment 4: Example Titration Benchsheet - LIMS

19.0 Revision History

- Revision 5, dated 31 March 2014
  - Removed Section 1.5, statement of total analytical time per sample
  - Revised Section 7.2. Removed reference to ASTM Type II water. DI water evaluated based on its resistivity measurement alone.
  - Revised Section 9.2.3 to remove reference to creating NCM for MS/MSD failure; data are flagged
  - Added Section 10.6, Troubleshooting and Maintenance
- Revision 4, dated 15 July 2013
  - Annual Technical Review
  - Corrected formatting and grammar errors
  - Changed the title to match source methods
  - Added Sections 5.3.4 and 5.3.5
  - Added Iodine safety information to the table in Section 5.4
  - Added TALS reagent/standard names to Section 7.0
  - Removed Potassium Iodide (KI) crystals from Section 7.0 – Iodine purchased

commercially
  - Removed Sulfuric Acid 20% from Section 7.0 due to it not being used
  - Added Sulfide crystals and Zinc acetate dehydrate to Section 7.0 – stock standards/reagents for recipes.
  - Revised MB criteria to reflect current practice
  - Added clarification to Section 10.5.1.7 for titration procedure
  - Updated Section 12.1 to reflect current practice
  - Updated Attachment 3 to match current validated spreadsheet

- Revision 3.3, dated 26 June 2012
  - Revised section 7 and references to standard solution
  - Revised references in section 8 to SW-846
  - Corrected Equation 1 (Section 11)
  - Updated method modifications for soil preservation, dynamic range, measurements for aqueous samples.
  - Source method review
  - Updated attachments
  - Formatting and grammatical changes throughout

- Revision 3.2, dated 31 May 2011
  - Annual Technical Review
  - Updated Attachments 2-4.

- Revision 3.1, dated 05 May 2010
  - Annual Review
  - Added section 6.1

- Revision 3, dated 27 March 2009
  - Incorporated method 9034 into this SOP
  - Fixed formatting and minor grammatical errors

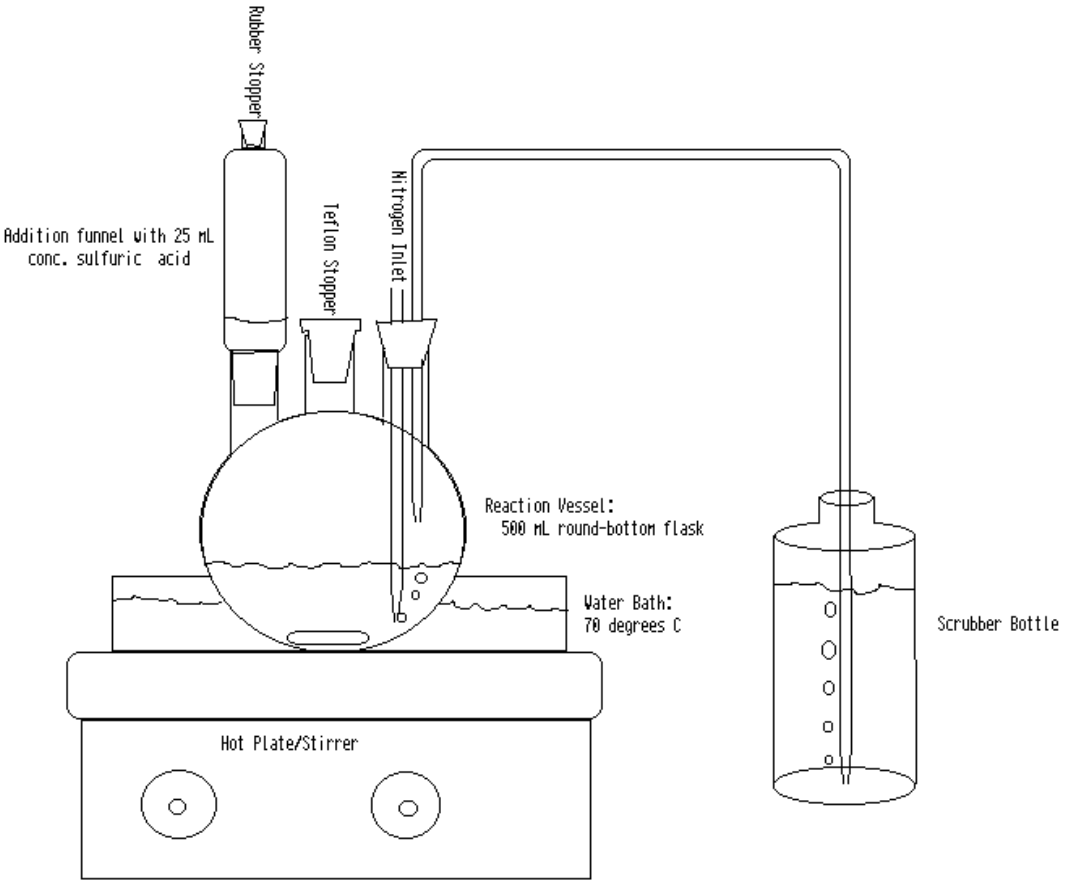
- Revision 2, dated 21 March 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting.
  - Updated method references.
  - Technical review performed.

- Revision 1.1, dated 21 November 2006
  - For this minor revision, the technical content of this SOP was not reviewed or updated. The purpose of this minor revision is to incorporate the Safety Bulletin information to ensure compliance with STL Corporate requirements. In addition, Section 9.1 was revised to reference Policy QA-024 for specific QC requirements for federal programs, any existing interim changes were incorporated, and formatting was updated consistent with Policy QA-001.

- Revision 1, dated 07 March 2002
  - Quanterra changed to STL.
  - Added Data review checklist to Appendix III.
  - Updated waste section.

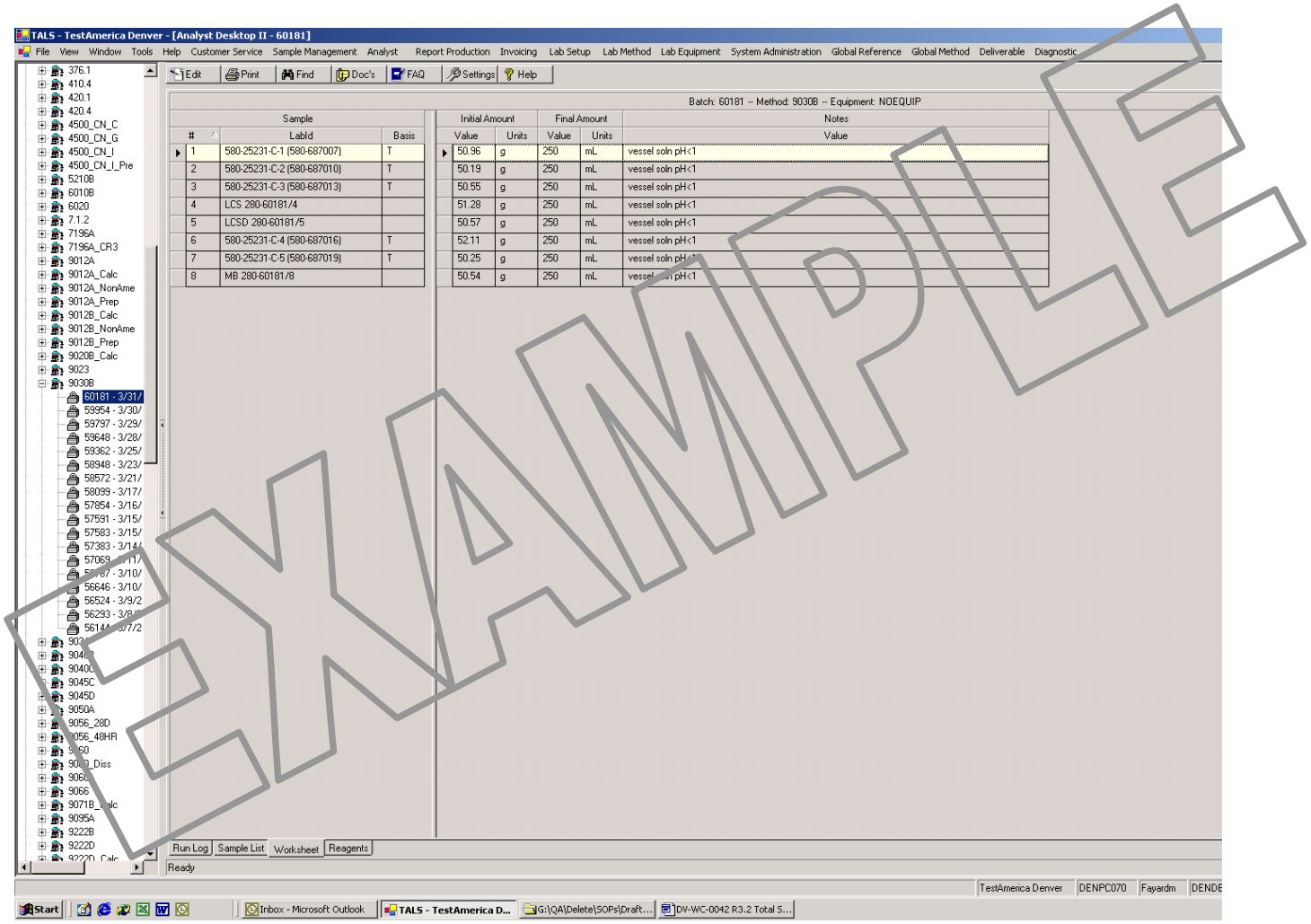
Attachment 1.

Distillation Apparatus



Attachment 2.

Example Sample Preparation Benchsheet - LIMS



Attachment 3.
Example Titration Benchsheet – Excel Worksheet

Laboratory Bench Sheet

TestAmerica-Denver

Sulfide by Titration

Analyst:	PM Plumb	Titration Solutions		SOP Information:		Rep. / Detection Limits	
	5/2/2011	Solution 1:	Iodine	Number:	DV-WC-44	MDL	0.7[illegible]
		Source:	Fisher	Revision:	4.3	Rep. Limit	4[illegible]
		Lot #:	0599-11	Calibration Information			
ICV Information		Normality:	0.025	Source/Ver-Lot#:	SFD CAL 275		
Source/Ver-Lot#:		Exp. Date:	1231/2011	Prep Date:	3/7/2011	Starch Indicator	
Prep Date:		Solution 2:	sodium thiosulfate	Made By:	pmp	La[illegible] Chem	
Made By:		Source:	Fisher	Concentration:	9[illegible]4	0600-[illegible]1	
Concentration:		Lot #:	0601-11	Expiration Date:	6/[illegible]/2011	expires:	4/26/2012
Expiration Date:		Normality:	0.025	LCS True Value:	1[illegible]68		
ICV True Value:		Exp. Date:	4/31/2012	Spike True Value:	19.[illegible]8		

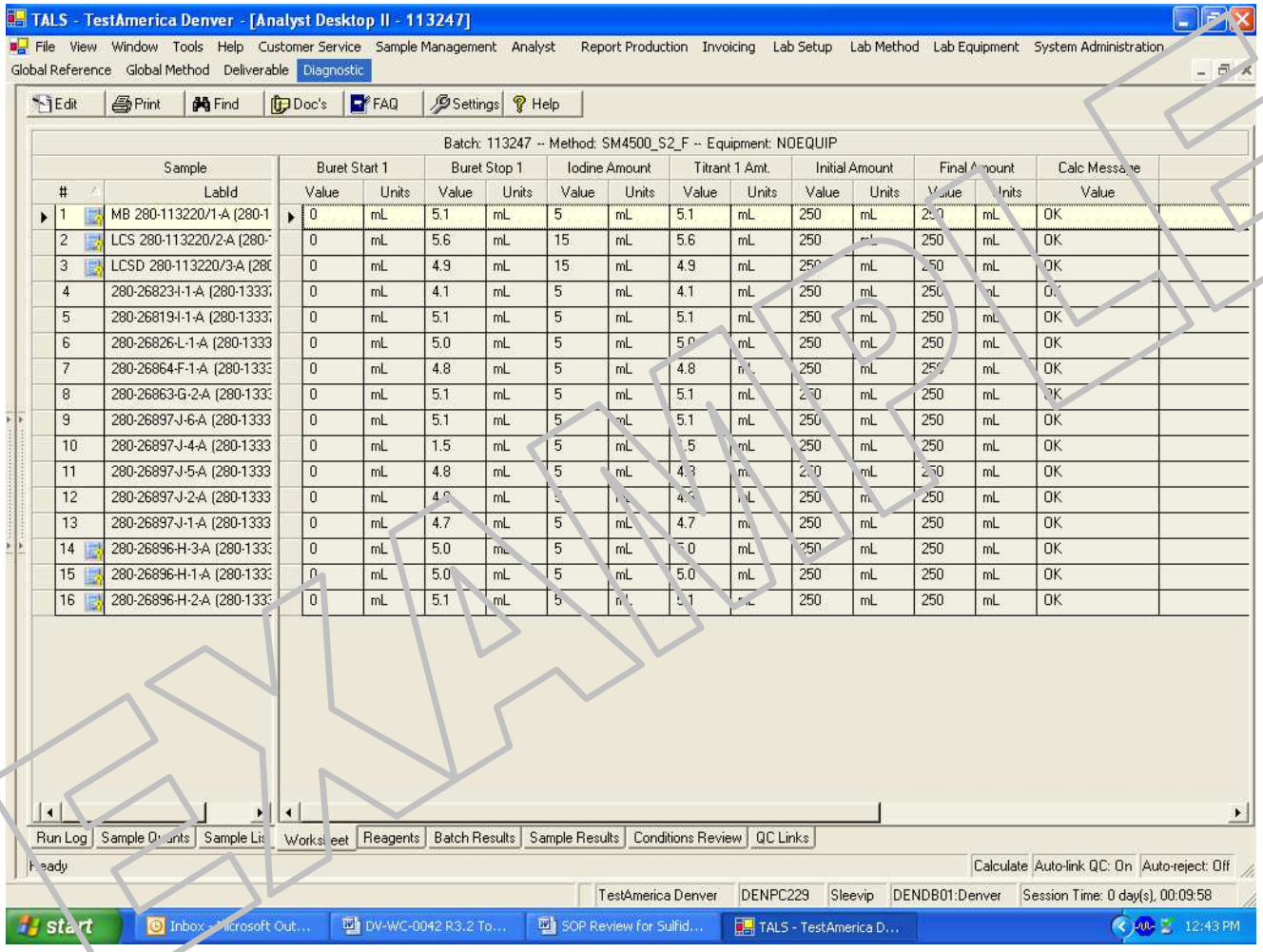
Lot Number	Work Order	Sample	Buret Start	Buret	mL Iodine	Final mL	Concentatn mg/L	Prep D.F.	Analyt. D.F.	Final Conc mg/L	% Rec.
	CAL	5	0.00	7.70	20	7.70	984.000	1	1	984.00	
	CAL	5	0.00	7.70	20	7.70	984.000	1	1	984.00	
	MB	250	0.00	5.10	5	5.10	-0.160	[illegible]	[illegible]	ND	
LCS	DCS	250	0.00	6.[illegible]0	15	[illegible]	13.760	1	[illegible]	13.76	69.92
LCSD	DCS	250	0.00	6.00	15	6.00	14.400	1	1	14.40	73.17
15033-1		250	0.00	4.60	5	4.60	[illegible]	1	1	ND	
15033-2		250	0.00	4.70	5	4.70	0.480	[illegible]	1	ND	
15033-3		250	0.00	5.00	5	5.00	0.000	1	1	ND	
15081-1		250	0.00	4.10	5	4.10	1.440	1	1	1.44	
15081-2		250	0.00	4.40	5	4.40	0.960	[illegible]	1	0.96	
15086-1		250	0.00	4.40	5	4.40	0.960	1	1	0.96	
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		
		250	0.00		5			1	1		

EXAMPLE

G:\QA\Edit\FORMS\Wet Chemistry\Sulfide\Sulfide by Titration_Master.xls
Revision 4 - March 2011

Attachment 4.

Example Titration Benchsheet - LIMS



#	Sample LabId	Buret Start 1 Value	Buret Start 1 Units	Buret Stop 1 Value	Buret Stop 1 Units	Iodine Amount Value	Iodine Amount Units	Titrant 1 Amt. Value	Titrant 1 Amt. Units	Initial Amount Value	Initial Amount Units	Final Amount Value	Final Amount Units	Calc Message Value
1	MB 280-113220/1-A (280-1	0	mL	5.1	mL	5	mL	5.1	mL	250	mL	250	mL	OK
2	LCS 280-113220/2-A (280-	0	mL	5.6	mL	15	mL	5.6	mL	250	mL	250	mL	OK
3	LCSD 280-113220/3-A (280	0	mL	4.9	mL	15	mL	4.9	mL	250	mL	250	mL	OK
4	280-26823-I-1-A (280-1333	0	mL	4.1	mL	5	mL	4.1	mL	250	mL	250	mL	OK
5	280-26819-I-1-A (280-1333	0	mL	5.1	mL	5	mL	5.1	mL	250	mL	250	mL	OK
6	280-26826-L-1-A (280-1333	0	mL	5.0	mL	5	mL	5.0	mL	250	mL	250	mL	OK
7	280-26864-F-1-A (280-1333	0	mL	4.8	mL	5	mL	4.8	mL	250	mL	250	mL	OK
8	280-26863-G-2-A (280-1333	0	mL	5.1	mL	5	mL	5.1	mL	250	mL	250	mL	OK
9	280-26897-J-6-A (280-1333	0	mL	5.1	mL	5	mL	5.1	mL	250	mL	250	mL	OK
10	280-26897-J-4-A (280-1333	0	mL	1.5	mL	5	mL	1.5	mL	250	mL	250	mL	OK
11	280-26897-J-5-A (280-1333	0	mL	4.8	mL	5	mL	4.8	mL	250	mL	250	mL	OK
12	280-26897-J-2-A (280-1333	0	mL	4.9	mL	5	mL	4.9	mL	250	mL	250	mL	OK
13	280-26897-J-1-A (280-1333	0	mL	4.7	mL	5	mL	4.7	mL	250	mL	250	mL	OK
14	280-26896-H-3-A (280-1333	0	mL	5.0	mL	5	mL	5.0	mL	250	mL	250	mL	OK
15	280-26896-H-1-A (280-1333	0	mL	5.0	mL	5	mL	5.0	mL	250	mL	250	mL	OK
16	280-26896-H-2-A (280-1333	0	mL	5.1	mL	5	mL	5.1	mL	250	mL	250	mL	OK

EXAMPLE

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Title: Total and Amenable Cyanide by SM 4500-CN⁻ B, 4500-CN⁻ C, 4500-CN⁻ E, 4500-CN⁻ G, SW 9012A and SW 9012B Weak Acid Dissociable Cyanide by 4500-CN⁻ I

Approvals (Signature/Date):

4/8/14	Adam W Alban 09 april 14
Richard Clinkscales — Date	Adam Alban — Date
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4/9/14	4/9/14
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1.0 Scope and Application

1.1 Analytes, Matrices, and Reporting Limits

1.1.1 This procedure is for the determination of:

Analyte	CAS Number	Reporting Limit Water (mg/L)	Reporting Limit Soil (mg/Kg)
Total Cyanide	57-12-5	0.01	0.50
Amenable Cyanide	STL00015	0.01	0.50
Weak Acid Dissociable Cyanide	STL00195	0.01	0.50
Free Cyanide	STL00131	0.01	0.50

1.1.2 This procedure is applicable to drinking, ground, surface and saline waters, solids and wastes.

1.1.3 This procedure detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine total cyanide, amenable cyanide and weak acid dissociable cyanide as described in Standard Methods 4500-CN^- B, 4500-CN^- C, 4500-CN^- E, 4500-CN^- G and 4500-CN^- I and total and amenable cyanide by SW-846 Methods 9012A and 9012B.

1.1.4 Cyanide amenable to chlorination and weak acid dissociable cyanide appear to be identical, however, in some industrial effluents the cyanide amenable to chlorination yields negative values and the measure of weak acid dissociable is a better alternative.

1.1.5 This procedure describes the reduced volume version of the methods and uses the same reagents and molar ratio to meet the quality control and performance requirement stated in the method.

1.1.6 Dynamic Range

The approximate working range extends from 0.01 mg/L to 0.4 mg/L for water samples and 0.5 mg/kg to 20 mg/kg for soil samples. Samples with higher concentrations are analyzed with dilution. Current method detection limits are given in the laboratory LIMS system.

2.0 Summary of Method

2.1 Total cyanide – Cyanide, as hydrocyanic acid (HCN) is released from samples by means of reflux-distillation under acidic conditions and absorbed in a scrubber containing sodium hydroxide (NaOH) solution.

2.2 The cyanide concentration in the scrubber solution is determined using an automated analyzer. The cyanide is converted to cyanogen chloride by reactions with Chloramine-T at pH <8 that subsequently reacts with pyridine and barbituric acid to give a red-colored complex. The color intensity which is proportionate to the cyanide concentration is measured at 570 nm. The concentration of NaOH must be the same in the standards, the scrubber solutions and any dilution of the original scrubber solution.

2.3 Cyanide amenable to chlorination is determined by using two sample aliquots. The first aliquot is distilled for total cyanide and the second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination as described above. Cyanide amenable to chlorination is the difference in these two values.

2.4 Weak acid dissociable cyanide is determined by distillation of the sample to which has been added acetate buffer and zinc acetate solution and sufficient acetic acid to obtain a pH of approximately 4.5-6 as determined by methyl red indicator. The cyanide concentration collected under these conditions is determined as described in Section above.

3.0 Definitions

3.1 Cyanide: The term "cyanide" refers to all of the CN groups in cyanide compounds that can be determined as the cyanide ion, CN^- by the various chemical methods. These compounds include both simple and complex cyanides.

3.2 Total Cyanide: All cyanides including nondissociable cyanides and cyanide bound in complexes that are readily dissociable or of intermediate stability

3.3 Cyanide Amenable to Chlorination: Free cyanide and complex cyanides that are potentially dissociable, almost wholly or in large degree.

3.4 Weak Acid Dissociable Cyanide: Cyanide that is available in a slightly acidified solution, including free cyanide.

3.5 Free Cyanide: Cyanide that is not complexed in the sample. Free cyanide is determined as weak acid dissociable cyanide.

4.0 Interferences

4.1 Oxidizing agents such as chlorine will destroy cyanide. Ascorbic acid is used to remove chlorine interferences.

4.2 Some unidentified organic compounds may oxidize or form decomposition products during chlorination, giving higher results for cyanide after chlorination than before chlorination; this gives a negative value for cyanide amenable to chlorination. The weak acid dissociable method should be used for these samples.

4.3 Samples that contain sulfide compounds may produce hydrogen sulfide during the distillation and interfere with color development. This is treated by adding

cadmium chloride (Standard Methods) or bismuth nitrate (SW-846 methods) to the sample prior to distillation, which removes sulfur by precipitation as cadmium sulfide or bismuth sulfide.

NOTE: Requirements for sample and standard processing are different for Standard Methods or SW-846 Methods. See Section 10.3.2.

4.4 Chlorine added to the sample for amenable cyanide must be completely destroyed before distillation. Otherwise, it may distill over and destroy the non-amenable cyanide.

4.5 Nitrate and/or nitrite may react with organic compounds during distillation to form cyanide. Sulfamic acid is added to remove the nitrate and/or nitrite interference.

4.6 Samples containing surfactants may foam excessively during distillation.

4.7 High carbonate concentrations may react violently when sulfuric acid is added to the samples during distillation.

4.8 Aldehydes, glucose, and other sugars may convert cyanide to cyanohydrin during distillation. If the client has indicated the possible presence of aldehydes, add 3.5% ethylenediamine as described in Section 10.3.3.

4.9 Amino acids may distill with the cyanide and interfere with the analysis.

4.10 Fatty acids may interfere by forming soaps in the absorption solution.

4.11 Thiocyanate greater than 10 mg/L may interfere.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, latex or nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Potassium cyanide and sodium cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in**

the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, and dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Potassium Hydroxide	Corrosive Poison Reactive	2 mg/m^3 - ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting and diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness and swelling. Greater exposures cause severe burns with possible blindness.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hydrochloric Acid	Corrosive Poison	5 ppm - ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat and upper respiratory tract and in severe cases, pulmonary edema, circulatory failure and death. Can cause redness, pain and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric acid	Corrosive Poison Irritant, Carcinogen	1mg/m^3 TWA	Inhalation symptoms may include irritation of the nose and throat, and labored breathing. Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death. Can cause sore throat, vomiting, and diarrhea. Skin contact can cause redness, pain, and severe burn. Eye contact can cause blurred vision, redness, pain and severe tissue burns.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Calcium hypochlorite	Strong oxidizer	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Glacial acetic acid	Corrosive Poison Flammable Irritant	10 ppm TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Swallowing can cause severe injury leading to death. Skin contact may include redness, pain, and skin burns. Eye contact may cause severe eye damage followed by loss of sight.
Sulfamic acid	Corrosive Irritant	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Chloramine-T	Irritant	None listed	May cause irritation to the mucous membranes and upper respiratory tract, skin and eyes.
Barbituric acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as a potential health hazard; do not ingest.
Bismuth nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m^3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns, and eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5 Build-up of pressure in the distillation apparatus will cause the hot, acidic solution to spray out of the thistle tube. In case vacuum is lost, the condensers must be opened to prevent build-up of pressure. If the solution overflows onto the heating block, turn it off.

5.6 All distillations are to be performed with adequate ventilation.

5.7 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.8 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit. For cyanide amenable to chlorination, the chlorination step will also be performed in a fume hood.

5.9 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Denver associate. The situation must be reported immediately to a laboratory supervisor and the Health and Safety Officer.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Alpkem Automated Segmented Flow Analyzer

- Autosampler
- Proportioning pump
- Injection module
- Colorimeter with 570 nm filter and 10 mm flow cell
- WinFlow Software
- Debubblers
- Miscellaneous tubing and reaction coils.

6.1.2 Midi-distillation apparatus consisting of 100 mL distillation tubes, cold-finger condensers, absorption tubes, and associated apparatus.

6.1.3 Vacuum pump.

6.1.4 Recirculating chiller.

6.2 Supplies

6.2.1 Disposable auto-sampler vials or culture tubes for samples.

6.2.2 Syringe filters with 0.45 µm filter.

6.2.3 Eppendorf pipettes, various sizes.

6.2.4 Volumetric flasks, class A, various sizes.

6.2.5 Volumetric pipettes, class A, various sizes.

6.2.6 Miscellaneous laboratory apparatus (e.g., magnetic stirrer) and glassware.

6.2.7 pH test strips.

6.2.8 Lead acetate test paper.

6.2.9 Potassium iodide-starch test paper.

6.2.10 Boiling chips.

6.3 Computer Software and Hardware

Please refer to the master list of documents, software, and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

NOTE: TALS IDs for standards and reagents are given in parentheses.

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Cyanide Calibration Stock Standard, 1,000 mg/L (CN CAL STD_)

7.2.1 This standard is purchased commercially.

7.2.1.1 It is not necessary to standardize the commercial product if it is accompanied by a certificate of analysis.

7.2.1.2 All intermediate and working standard concentrations are adjusted from the nominal concentrations shown below to the exact concentrations based on the certified concentration from the vendor.

7.2.2 *Alternatively,* the calibration stock standard can be made as described here.

7.2.2.1 Dissolve 2.51 g of dried (103°C) potassium cyanide and 2.0 g potassium hydroxide in water and dilute to 1,000 mL.

7.2.2.2 This stock solution (prepared by the lab) will be standardized initially and every 30 days thereafter as described in Section 7.3.

7.2.2.3 All intermediate and working standard concentrations are adjusted from the nominal concentrations shown below to the exact concentrations based on the results of the monthly standardization.

7.3 Standardization of the Stock Cyanide Solution (if No Certificate of Analysis Available)

7.3.1 Reagents:

7.3.1.1 Indicator: Dissolve 20 mg p-dimethylaminobenzalrhodanine in 100 mL acetone.

7.3.1.2 Silver nitrate ($AgNO_3$) titrant; 0.0192 N: Obtain a commercially prepared certified standard.

7.3.2 Cyanide Standardization

7.3.2.1 Add 10 mL of the 1000 mg/L Cyanide Stock Standard (Section 7.2.2) solution to a 500 mL Erlenmeyer flask. Dilute to 250 mL with deionized water.

7.3.2.2 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution.

7.3.2.3 Titrate with standard silver nitrate, $AgNO_3$, to the first change in color from a canary yellow to a salmon hue.

7.3.2.4 Prepare a blank in a similar fashion. Add 250 mL of deionized water to a 500 mL Erlenmeyer flask.

7.3.2.5 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution. Titrate with standard $AgNO_3$ to the first change in color from a canary yellow to a salmon hue. The standardization should be done in duplicate.

7.3.2.6 Calculate the true stock cyanide concentration as follows:

$$mg/L\ Stock\ Cyanide = \frac{[(A-B)\times 1000]}{mL\ of\ sample}$$ *Equation 1*

Where:

A = Volume of $AgNO_3$ for titration of sample, mL
B = Volume of $AgNO_3$ for titration of blank, mL

7.3.2.7 If the verification result is within 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard the LIMS Reagent module.

7.3.2.8 If the verification result is less than 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard in the LIMS Reagent module.

7.3.2.8.1 Create a “new” standard in the LIMS Reagent Module for this standard using the new concentration from the verification process.

7.3.2.8.2 Prepare a label for the stock solution with the name of the analyst, date of verification, expiration date (one month beyond the date of re-standardization), lot number, 1% (0.25 N) NaOH, and the actual stock cyanide concentration.

7.3.2.9 The true concentration of the stock cyanide solution after standardization with silver nitrate is used to prepare intermediate and working standards. This concentration does not always equal 1000 mg/L. The dilution factor relative to the stock standard is provided to aid in the calculations to determine the True Working Standard concentration for the standards listed in Section 7.5.2, where:

$$Conc.\ of\ True\ Working\ Std = \frac{True\ Conc.\ of\ Stock}{DF}$$ *Equation 2*

Where:

$$DF = \frac{Vol.\ of\ intermed\ std\ used\ (mL)}{Final\ Vol\ of\ dilution\ (mL)}$$ *Equation 3*

7.4 Intermediate Standard I, 10 mg/L (CN 10ppm_)

7.4.1 Obtain a clean 100 mL class A volumetric flask.

7.4.2 Add approximately 50 mL of the 1% (0.25 N) sodium hydroxide (1% NaOH) to the flask.

7.4.3 Pipette 1.0 mL of the 1,000 mg/L calibration stock standard (Section 7.1) into the flask.

7.4.4 Dilute to volume with 1% (0.25 N) sodium hydroxide (1% NaOH). Prepare every 7 days.

7.5 Calibration Standards

7.5.1 Working Standard, 1.0 mg/L (CN CAL 1 ppm)

7.5.1.1 Pipette 10.0 mL of Intermediate Standard I (Section 7.4) into a 100 mL volumetric flask.

7.5.1.2 Dilute to volume with 1% (0.25 N) sodium hydroxide and mix. Prepare daily.

7.5.2 Initial Calibration Standards

Dilute the 1.0 mg/L or 0.1 mg/L cyanide working standard with 1% (0.25 N) sodium hydroxide as follows:

Standard Level	Intermediate	Vol of Std Used (mL)	Final Volume (mL)	Concentration (mg/L)
1	N/A	0	50	0.00 (Blank)
2	1 mg/L	0.5	50	0.01
3	1 mg/L	1.0	50	0.02
4	1 mg/L	2.5	50	0.05
5	1 mg/L	5.0	50	0.10
6	1 mg/L	10.0	50	0.20
7	1 mg/L	10.0	25	0.40

Calculate the exact concentration for each calibration curve standard using Equations 2 and 3 if stock concentration is different from 1.0 mg = 1.0 mL.

When using the LIMS Reagent Module, the concentration of the calibration standards will be automatically calculated.

7.6 Continuing Calibration Verification Standard (CCV), 0.2 mg/L:

The Level 5 calibration standard described in Section 7.5.2 is used as the working CCV standard.

7.7 Second-Source Standards

7.7.1 Initial Calibration Verification (ICV) Stock Standard, 1,000 mg/L (CN ICV Std_)

The second-source standard is obtained from a different source than the calibration standards. This standard is available commercially. Manufacturer's expiration date is used. It is prepared and standardized as described in Section 7.3 for the primary standard if unavailable commercially. Standardization is only required if no Certificate of Analysis is available for the standard.

7.7.2 Intermediate ICV (second-source) Standard, 10 mg/L (CN ICV Int_)

Pipette 1.0 mL of the 1,000 mg/L ICV stock (Section 7.7.1) into a 100 mL volumetric flask. Dilute to volume with 1% (0.25 N) sodium hydroxide. Prepare every 7 days.

7.7.3 Working ICV (second-source) Standard, 0.10 mg/L (CN ICV Daily_)

Spike 1 mL of the 10 mg/L intermediate (Section 7.7.2) into a 100 mL volumetric flask and fill to the mark with 1% (0.25 N) NaOH. Prepare daily.

7.8 Pyridine-Barbituric Acid Solution, per OI Manual (CN PYR/BARB_)

7.8.1 In a hood, place 15 g barbituric ($C_4H_4N_2O_3$) acid (Barbititric_) in a 1000 mL volumetric flask and add about 100 mL deionized water, rinsing down the sides of the flask.

7.8.2 Place on a magnetic stirrer and add a stir bar.

7.8.3 Add 75 mL pyridine (Pyridine_) while mixing.

7.8.4 Carefully add 15 mL concentrated hydrochloric acid (HCL_) while mixing.

7.8.5 Add 500 mL of DI water and stir until the barbituric acid is dissolved.

7.8.6 Dilute to volume with deionized water.

7.8.7 Store in an amber bottle.

7.8.8 Expires 6 months from preparation.

7.9 Phosphate Buffer Solution 1 M, per OI Manual (CN BUFFER_)

7.9.1 Dissolve 138 g sodium dihydrogen phosphate monohydrate ($NaH_2PO_4 \bullet H_2O$) (Sodium Phos_) in deionized water and dilute to 1000 mL.

7.9.2 Add 4 mL of Brij-35 to the solution and mix gently.

NOTE: Actual volume varies depending upon equipment operation. It may be necessary to add additional Brij-35 for smooth operation of the equipment.

7.9.3 Store at room temp.

7.9.4 Filter the solution through a glass fiber filter.

7.9.5 This solution expires 3 months from preparation.

7.10 Chloramine-T, per OI Manual (CN CHLOR-T_)

7.10.1 Dissolve 2.0 g Chloramine-T in deionized water and dilute to 250 mL.

7.10.2 Prepare fresh daily.

7.11 Sodium Hydroxide, 10 N (10N_NaOH_), *Commercial Solution Purchased*

7.11.1 Dissolve 400 g sodium hydroxide in deionized water.

7.11.2 Cool to room temperature, dilute to 1000 mL, and mix well.

7.11.3 Store in a plastic bottle.

7.12 Sodium Hydroxide, 2% wt/wt (0.5 N) (2% NaOH_)

7.12.1 Place 80 g NaOH in a plastic 4 L container.

7.12.2 Add 2000 mL of DI water to the container.

7.12.3 Add a magnetic stir bar and stir until the NaOH is dissolved.

7.12.4 Add 2000 mL of DI water to the flask. Stir to mix.

7.13 Sodium Hydroxide Dilution Solution, 1% wt/wt (0.25 N) (1% NaOH_)

7.13.1 Place 20 g of NaOH in a 2L volumetric flask.

7.13.2 Add approximately 1L of DI water.

7.13.3 Add a magnetic stir bar and stir until the NaOH is dissolved.

7.13.4 Allow to cool, remove stir bar, bring to volume with DI, and mix well.

7.14 Sulfuric acid, concentrated, reagent grade (H2SO4_)

7.15 Sulfuric acid, 0.02 N (0.02 H2SO4_) *Commercial Solution Purchased*

7.15.1 If commercial solution is not available, this solution can be prepared as follows. In a 2000 mL volumetric flask, carefully add 1 mL concentrated sulfuric acid to approximately 1900 mL deionized water.

7.15.2 Dilute to final volume of 2000 mL with deionized water and mix.

7.16 Bleach

Fragrance free commercial liquid bleach is purchased and used in place of the calcium hypochlorite solution. Replace 1 month after opening.

7.17 Magnesium Chloride solution, 2.5 M (CN Mag Chl_)

This reagent is purchased though an approved vendor.

7.18 Glacial acetic acid, reagent grade. (Acetic acid_)

7.19 Acetate buffer (WAD Acetate_)

7.19.1 Dissolve 410 g sodium acetate trihydrate (SODIUM ACETAT_) in approximately 450 mL deionized water.

7.19.2 Adjust the pH to 4.5 with glacial acetic acid and dilute to final volume of 500 mL with deionized water.

7.19.3 The solution expires 1 year from preparation.

7.20 Zinc Acetate solution (Zinc Buffer_)

7.20.1 Dissolve 100 g zinc acetate dihydrate in a 1 liter volumetric flask filled with approximately 500 mL deionized water.

7.20.2 Dilute to final volume of 1000 mL with deionized water.

7.20.3 Store in a 1 L plastic container.

7.20.4 The solution expires 1 year from preparation.

7.21 Methyl Red Indicator solution (Methyl Red_)

7.21.1 Dissolve 0.1 g methyl red in 100 mL deionized water.

7.21.2 Expires 1 year from preparation.

7.22 Acetic acid, 10% (10% Acetic Acid_)

7.22.1 In a vent hood, carefully add 100 mL glacial acetic acid to about 500 mL deionized water, mix, and dilute to 1000 mL.

7.22.2 The solution expires 1 year from preparation.

7.23 Ascorbic acid crystals (Ascorbic Acid_)

7.24 Sulfamic Acid (NH_2SO_3H), 10% wt/wt (CN SULFAMIC_)

7.24.1 Dissolve 100 g sulfamic acid in 1000 mL of deionized water. Mix well.

7.24.2 Store in a repipetter container.

7.24.3 Expires 1 year from preparation.

7.25 Cadmium Chloride powder, ($CdCl_2$), (CAD CHL CN_)

7.25.1 Used to remove sulfide interference.

7.26 3.5% Ethylenediamine (EDTA) solution

7.26.1 Used to remove client identified aldehyde interferences

7.27 Bismuth Nitrate ($Bi(NO)_3 \cdot 5H_2O$), 0.062 M, (CN_BiN3O9_)

7.27.1 Obtain a clean, dry 250 mL volumetric flask.

7.27.2 Add approximately 75 mL of DI water to the flask.

7.27.3 Add 7.5 g bismuth nitrate to the flask and stir to dissolve.

7.27.4 *Slowly* add 60 mL of glacial acetic acid, swirling frequently.

7.27.5 Stir until completely dissolved.

7.27.6 Bring to volume with DI water.

7.28 Brij-35 Start-Up Solution:

Concentrated Brij-35 is a buffer solution obtained from the equipment vendor. The start-up solution is prepared by diluting 1 mL of the Brij-35 concentrate to 500 mL with reagent water.

7.29 Reagent Water: Deionized water $\leq$ 1.0 µmho-cm.

7.30 Teflon boiling chips for use as solid matrix for LCS and MB. Record lot number in TALS in prep batch information.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water[1]	HDPE, Glass	500 mL	Cool, ≤ 6°C	48 hours **	40 CFR Part 136.3
Water[1,2]	HDPE, Glass	500 mL	NaOH, pH > 10; Cool, ≤ 6°C;	14 days	40 CFR Part 136.3
Water[2]	HDPE, Glass	500 mL	NaOH, pH > 12; Cool, ≤ 6°C	14 days	SW-846
Solid	HDPE, Glass	5 g	Cool, ≤ 6°C	14 days	SW-846

[1] Add 1.2 g of ascorbic acid per liter of sample if residual chlorine is present.

[2] Preservation to pH > 10 for NPDES compliance samples (Method 335.4, the SM 4500-CN^- methods); pH >12 for Methods 9012A and 9012B.

**** NOTE:** If the client sample arrives unpreserved the sample needs to be checked for interferences (Section 10.3). If there are interferences, treat the sample and preserve immediately. If there are no interferences preserve with NaOH (pH noted above) and follow the 14 day holding time. Document the preservation using an observation Nonconformance Memo (see SOP DV-QA-0033).

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This SOP meets the requirements of DoD QSM 5.0 unless otherwise specified.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as

appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Sample QC: The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	<1/2 Reporting Limit or < 10% of sample concentration
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Total Cyanide: .90-110% LCS/LCSD RPD: ≤ 10% Statistical Limits[4] for WAD
Matrix Spike (MS)[2]	1 in 10 or fewer samples	Total Cyanide: .90-110% LCS/LCSD RPD: ≤ 10% Statistical Limits[4] for WAD
MS Duplicate (MSD)[2]	1 in 10 or fewer samples	Total Cyanide: .90-110% MS/MSD RPD: ≤ 10% Statistical Limits[4] for WAD
High Distilled Standard (HLCS) (0.4 mg/L)	1 in 20 or fewer samples	± 10% of true value
Low Distilled Standard (LLCS) (0.10 mg/L)	1 in 20 or fewer samples	± 10% of true value

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are stored in the LIMS.

9.3 Method Blank

9.3.1 Water Samples:

Add 50 mL of 1% (0.25 N) NaOH into a distillation flask immediately prior to distillation.

9.3.2 Solid samples:

Weigh 1.0 g of Teflon chips into a distillation flask and add 50 mL 1% (0.25 N) NaOH.

Acceptance Criteria: Concentrations in the method blank must be less than one-half the reporting limit or less than 10% of the sample concentration.

Corrective Action: The corrective action for method blank failures is redistillation and reanalysis of all samples in the batch. If there is insufficient sample for reanalysis, a Nonconformance Memo (NCM)

must be prepared and the client contacted by the laboratory Project Manager.

9.4 LCS / LCSD (Second Source)

9.4.1 Water Samples

9.4.1.1 Measure 50 mL of 1% NaOH in a graduated cylinder and transfer to the distillation flask.

9.4.1.2 Spike 0.5 mL of the 10 mg/L second source intermediate standard (Section 7.7.2) into the flask. Swirl to mix.

9.4.2 Soil Samples

9.4.2.1 Weigh 1.0 g of Teflon chips into a distillation flask.

9.4.2.2 Measure 50 mL of 1% NaOH in a graduated cylinder and transfer to the distillation flask.

9.4.2.3 Spike with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.7.2) into the flask. Swirl to mix.

Acceptance Criteria: LCS recoveries for Total Cyanide are 90-110%. Recoveries for Weak Acid Dissociable Cyanide (WAD) are compared to the historical limits stored in the LIMS. See also Section 9.2.

For DoD QSM 5.0, QC limits for LCS are:

Water: 83-116%
Solid: 76-120%

The LCS for Cyanide Amenable to Chlorination should be 0% which demonstrates that the chlorination process has worked effectively. (This is not reported.)

If a LCSD is also analyzed the RPD must be within ± 10% for Total Cyanide and within historical limits stored in LIMS not to exceed ± 20% for WAD. See Also Section 9.2.

Corrective Action: If the LCS fails, redistill and reanalyze all samples in the batch. If reanalysis is not possible, a Nonconformance Memo must be prepared and the client contacted by the laboratory Project Manager. See the TestAmerica Denver Policy, Quality Assurance Program, DV-QA-003P for additional guidance.

9.5 MS/MSD

9.5.1 Water Samples

Measure 25 mL of sample into a class A graduated cylinder. Spike the aliquot with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.7.2) and bring to 50 mL with sample. The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

9.5.2 Soil Samples

Weigh 1.0 g of sample into a distillation flask. Spike the sample aliquot with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.7.2), then add 1% (0.25 N) NaOH for a total volume of 50 mL. The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

Acceptance Criteria: MS/MSD recoveries are compared to the limits stored in the LIMS. See also Section 9.2. For QSM 5.0 the recovery limits are the same as the LCS (see section 9.4.2.3) and the RPD limit is ± 20%

Corrective Action: Because MS/MSD results may not have a direct bearing on other samples in the batch, the appropriate corrective action is generally governed by specific project requirements. At a minimum, QC failures will be noted as anomaly and discussed in the final report.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Any samples that have target analytes at such low levels do not provide useful precision data via duplicate analyses. If a sample duplicate is performed for DoD QSM 5.0, the RPD limit between duplicates is ± 20%.

9.6 High and Low Distilled Standards (HLCS & LLCS), 0.4 mg/L and 0.10 mg/L

Standards are distilled to monitor the efficiency of the distillation process and verify the linearity of the curve.

9.6.1 Water Samples

9.6.1.1 In each of two graduated cylinders, add 25 mL of 1% (0.25 N) NaOH.

9.6.1.1.1 For the HLCS, add approximately 25 mL of 1% NaOH to the graduated cylinder and spike 2.0 mL of

the 10 mg/L cyanide standard (or equivalent) and bring to 50 mL with 1% (0.25 N) NaOH.

9.6.1.1.2 For the LLCS, add approximately 25 mL of 1% NaOH to the graduated cylinder and spike 0.5 mL of the 10 mg/L cyanide standard (or equivalent) and bring to 50 mL with 1% (0.25 N) NaOH.

9.6.2 Soil Samples

9.6.2.1 Weigh 1 g of Teflon chips into a distillation flask for each standard.

9.6.2.1.1 For the HLCS, add 25 mL of 1% NaOH to the distillation flask and spike 2.0 mL of the 10 mg/L cyanide standard to the flask and add 25 mL of 1% NaOH for a total volume of 50 mL.

9.6.2.1.2 For the LLCS add 25 mL of 1% NaOH to the distillation flask and spike 0.5 mL of the 10 mg/L cyanide standard to the flask and add 25 mL of 1% NaOH for a total volume of 50 mL.

Acceptance Criteria: Recoveries for the HLCS and LLCS standards must be ± 10% of the true value. For DoD QSM 5.0, check method comments to determine if the variance for the LLCS is accepted, in which case the acceptance limits are ± 15% of the true value for LLCS and ± 10% for the HLCS.

Corrective Action: Distilled standard failure results in re-distillation and reanalysis of all associated samples. One possible exception is the situation in which recoveries are greater than 110% and cyanide was not detected in the samples. In that case, a Nonconformance Memo should be prepared and the failure noted in the report together with the sample results without taking other corrective action. For DoD QSM 5.0 check method comments to determine if this is acceptable for the project.

9.7 Instrument QC

9.7.1 Initial Calibration Verification (ICV)/ Initial Calibration Blank (ICB)

Immediately after the initial calibration, the calibration is verified using a second-source ICV standard and an initial calibration blank ICB (1% NaOH).

Acceptance Criteria: The measured result for the ICV must be within 10% of the expected value.

The ICB must be less than the reporting limit.

For DoD QSM 5.0, the ICB must be < LOD.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

9.7.2 Continuing Calibration Verification (CCV) / Continuing Calibration Blank (CCB)

A blank CCB (1% NaOH) and standard check CCV (see preparation in Section 7.6) are required after every 10 or fewer samples and at the end of the run.

Acceptance Criteria: The standard check (CCV) must be within 10% of the expected value.

Blanks must be less than the reporting limit.

For DoD QSM 5.0, the CCB must be < LOD.

Corrective Action: If either continuing calibration check fails, all samples since the last successful calibration check must be reanalyzed.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

10.3.1 The following sections describe separate preparation procedures for the different forms of cyanide.

Section 10.4: Cyanide Amenable To Chlorination
Section 10.5: Total Cyanide
Section 10.6 Weak Acid Dissociable Cyanide

10.3.2 Check aqueous samples for sulfide prior to distillation using lead acetate paper.

10.3.2.1 Moisten the paper with 2 or 3 drops of acetate buffer, and then place 1 drop of sample on the paper. A dark color indicates a positive test for sulfide. Record the result as "positive" or "negative" in the TALS prep batch.

10.3.2.1.1 If the test for sulfide is positive, Method 9012A and Method 9012B require that the samples be treated with bismuth nitrate rather than cadmium chloride and processed in a separate batch. The standards must be treated in the same manner as the samples, including addition of bismuth nitrate and distillation. See Section 10.5.9**.**

10.3.2.1.2 If the samples test positive using lead acetate paper and are to be analyzed using Standard Methods, treat 75 mL of the stabilized sample (pH > 12) with cadmium chloride.

10.3.2.2 Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper.

10.3.2.3 Filter the solution through a dry filter paper and from the filtrate, measure the sample aliquot to be used for analysis.

10.3.2.4 The filter papers are contaminated with cadmium. Use the designated disposal container for these filters.

10.3.3 If the client has specifically indicated that aldehydes may be present, 2 mL of 3.5% ethylenediamine is added per 100 mL of sample upon receipt.

10.4 Cyanide Amenable To Chlorination Sample Preparation

10.4.1 Two sample aliquots are required for the determination of cyanide amenable to chlorination. The first aliquot is distilled for total cyanide (see Section 10.5). The second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination.

NOTE: If the results for Total Cyanide are less than the reporting limit, the cyanide amenable to chlorination is not determined and reported as ND. The non-amenable cyanide is then equal to the total cyanide result. The method blank reported in this case is the Total Cyanide method blank.

NOTE: The chlorination process must be performed in a fume hood.

10.4.2 Measure the sample aliquots to be chlorinated (including a method blank, LCS and sample duplicate) into 100 mL beakers covered with a large plastic tub. Keep beaker covered with wrapped watch glass. Alternative means of protecting samples from light may be used.

10.4.2.1 For water samples, use 50 mL of sample.

10.4.2.2 For soil samples, use 1.0 g of sample and add 50 mL 1% (0.25 N) NaOH.

10.4.2.3 Clearly label the samples with the proper identification and "chlorinated" as appropriate.

10.4.3 Check the pH of samples with pH test strips. Record the results on the bench sheet.

10.4.4 Adjust the pH of the samples in the beakers to between 11 and 12 with the 10 N sodium hydroxide solution.

10.4.5 Test the samples with KI-Starch paper and add bleach solution drop-wise to each sample while mixing (use a magnetic stirrer) until a positive test is obtained. A positive test is indicated by a blue or black color on the paper.

10.4.6 Maintain the excess chlorine level in the sample for 1 hour while keeping the pH of the samples between 11 and 12 with constant mixing (use a magnetic stirrer). Add bleach solution and sodium hydroxide as necessary.

10.4.7 After 1 hour, add 0.1 g portions of ascorbic acid crystals until a negative test is obtained with KI-Starch paper.

10.4.8 Add an additional 0.1 g of ascorbic acid crystals to the sample to ensure an excess of the reagent

10.4.9 Transfer the contents of the beakers into distillation flasks quantitatively, rinsing with deionized water.

10.4.10 Proceed to section 10.5.4 for the distillation process.

10.5 Total Cyanide Sample Preparation

10.5.1 Check the pH of aqueous samples with pH test strips. Record the results on the TALS bench sheet. If the sample pH is $\leq$ 12 for Methods 9012A or 9012B or $\leq$ 10 for SM 4500 methods, document the improper preservation with a NCM.

10.5.2 Check aqueous samples for oxidizing agents such as chlorine.

10.5.2.1 Place one drop of sample on a strip of potassium iodide (KI)-starch test paper. A blue color indicates the need for treatment.

10.5.2.2 Record the result as “positive” or “negative” on the benchsheet.

10.5.2.3 If a positive test is obtained, add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper.

10.5.2.4 Add an additional 0.1 g of ascorbic acid in excess.

10.5.3 Measure sample aliquots into the distillation flasks as follows:

10.5.3.1 For water samples use 50 mL of sample.

10.5.3.2 For solid samples use 1.0 g of sample and add 50 mL 1% (0.25 N) NaOH.

10.5.3.3 Prepare the batch QC samples as described in Section 9 (MB, LCS, MS/MSD, LLCS, HLCS)

10.5.4 Place 25 mL 2% sodium hydroxide into the absorption tubes.

10.5.5 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

10.5.6 Turn on the vacuum pump and ensure the chiller is on. Also be sure that the slot hood is operating.

10.5.7 Adjust the vacuum to provide a flow rate of approximately 2-3 bubbles per second (i.e., this is approximately 1/8-1/4 inch of foam in the scrubber) in the distillation flask.

10.5.8 Verify that there are no leaks in the system by observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

10.5.9 If the samples are logged for Method 9012A or 9012B and the test for sulfide is positive, all standards (minimum of five standards and blank), QC and samples must be processed in a separate batch.

10.5.9.1 Add 5 mL of 0.062 M bismuth nitrate solution (see Section 7.27) through the thistle tube to every standard, sample and QC sample in the analytical batch.

10.5.9.2 Samples designated for analysis using DoD QSM 5.0 where the project specifically requires MSA are analyzed using the method of standard addition utilizing a 2-point spike and calculating the sample concentration using the MSA data. Consult your Supervisor, a Technical Specialist, or the QA Manager before proceeding.

10.5.10 Add 2 mL of 10% sulfamic acid solution (Section 7.24) through the thistle tube. Allow to mix for 3 minutes.

10.5.11 Slowly and carefully, add 2.5 mL concentrated sulfuric acid through the thistle tube. Rinse the tube with a little deionized water and allow to mix for 3 minutes.

10.5.12 Add 2 mL of magnesium chloride solution (Section 7.17) and mix. If excessive foaming is observed, add additional magnesium chloride.

10.5.13 Turn on the heating mantles and heat the samples to boiling. While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

10.5.14 Allow samples to reflux for 1.5 hours by initiating the timer on the heating mantle.

10.5.15 After 1.5 hours of refluxing, allow the samples to cool for 15-30 minutes while air is flowing.

10.5.16 *While the vacuum is still on*, remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with deionized water.

Note: It is important to keep the vacuum on to prevent the distillate from being trapped inside the bubbler.

10.5.17 Remove the flasks and dispose of the contents as directed in Section 14.0. Residue not removed by this method must be scrubbed out.

10.5.18 Dilute the sodium hydroxide in the absorption tube to 50 mL with deionized water and store in labeled plastic vials.

10.5.19 Place each batch of distillates in a box and store the samples at 4℃ in the sample refrigerator until they are analyzed.

10.5.20 At the end of the day, turn off the vacuum.

10.5.21 Proceed to Section 10.7 for colorimetric analysis of the distillates.

10.6 Weak Acid Dissociable Cyanide in Water – Sample Preparation (SM 4500-CN I)

10.6.1 Measure the pH of water samples with a pH test strip. Record the results on the bench sheet. If the sample pH is less than 12, document the improper preservation with a NCM.

10.6.2 Measure 50 mL or an aliquot diluted to 50 mL into a distillation flask for each sample plus matrix spike and matrix spike duplicate. (See Section 9.5).

10.6.3 Prepare the batch QC samples as described in Section 9 (MB, LCS, LLCS, and HLCS)

10.6.4 Record the sample volume on the bench sheet.

10.6.5 Place 25 mL 2% (0.5 N) sodium hydroxide into the absorption tubes.

10.6.6 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

10.6.7 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

10.6.8 Adjust the vacuum to provide a flow rate of about 4 bubbles per second in the distillation flask, verifying that there are no leaks in the system by

observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

10.6.9 Add through the air inlet tube: 2 mL acetate buffer (Section 7.19), 2 mL zinc acetate solution (Section 7.20), and 2 or 3 drops of methyl red indicator. Rinse tube with about 2 mL deionized water and allow to mix.

10.6.10 If the solution is not pink, add 10% acetic acid drop-wise until a pink color persists, rinsing in with deionized water.

10.6.11 Turn on the controller and heat the samples to boiling. While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

10.6.12 Allow samples to reflux for 1.5 hours. Then allow the samples to cool for 15 minutes while air is flowing. Record start and end times for the distillation in the batch record.

10.6.13 *While the vacuum is still on*, remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with deionized water.

Note: It is important to keep the vacuum on to prevent the distillate from being trapped inside the bubbler.

10.6.14 Dilute the sodium hydroxide in the absorption tube to 50 mL with deionized water and store in plastic vials.

10.6.15 Place each batch of distillates in a box and store the labeled distillates at 4℃ until they are analyzed.

10.6.16 Raise the cold-finger condensers and rinse into the distillation flasks with de-ionized water. Dispose of the contents as directed in Section 14.0. Residue not removed by this method must be scrubbed out.

10.6.17 Rinse absorber tubes with deionized water.

10.6.18 At the end of the day, turn off the vacuum.

10.6.19 Proceed to Section 10.7 for colorimetric analysis of the distillates.

10.7 Instrument Set-Up

10.7.1 Verify that the 570 nm filter is installed.

10.7.2 Instrument Stabilization (per Alpkem manual)

10.7.2.1 Connect the reagent pump tubes to a reagent bottle containing the start-up solution (Section 7.27).

10.7.2.2 Start the pump, allowing the start-up solution to flow through the entire system.

10.7.2.3 Make sure that the flow cell of the detector is purged of all bubbles and the flow is stable and free from surging.

10.7.2.4 Once a stable flow is achieved, connect the reagent pump tubes to their respective reagent bottles, as shown in the schematic in Attachment 3.

10.7.2.5 Allow the reagents to flow through the entire system, then, once again, verify that the flow cell of the detector is purged of all bubbles.

10.8 Initial Calibration

10.8.1 Calibration is performed daily or each time the instrument is set up using the standards shown in Section 7.5 and the external standard method.

NOTE: The use of internal standards is not applicable for this spectrophotometric method.

10.8.2 A minimum of five standards and a blank are required for the calibration. The high standard in Section 7.5 may be dropped if needed and sample dilutions performed appropriately.

NOTE: If sulfide was detected during the sample preparation step and the samples are logged for 9012A or 9012B, then bismuth nitrate must be used to precipitate the sulfide. The method of standard additions spike must be prepared, and all calibration standards must be treated and distilled in the same manner as the samples, including the addition of bismuth nitrate. A minimum of five standards and a blank shall be distilled. Use the same calibration levels as shown in the table in Section 7.5.

10.8.3 The calibration function is calculated by least-squares linear regression. See Section 11.2

Acceptance Criteria: The correlation coefficient, r, must be $\geq$ 0.995 ($r^2 \geq 0.99$)and the absolute value of the intercept must be lower than one-half the response for the reporting limit.

Corrective Action: If the correlation coefficient is < 0.995 or the absolute value of the intercept is too large, locate and correct the problem and re-calibrate the instrument.

10.8.4 Assess the peak height of the synchronization (sync) standard.

Acceptance Criteria: The peak height of the sync standard should be ± 10% of pervious sync.

Corrective Action: If the peak height is < 150,000, the flow cell of the instrument must be cleaned (consult

manufacturer's instructions), and then the instrument must be recalibrated

10.8.5 Initial Calibration Checks

Immediately after the initial calibration, the calibration is verified using a second-source, initial calibration verification (ICV, see preparation in Section 7.7.3) standard and an initial calibration blank (ICB, 1% NaOH).

Acceptance Criteria: The measured result for the ICV must be within 10% of the expected value, and the ICB must be less than the reporting limit.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

10.8.6 Continuing Calibration Checks

A standard check (CCV; see preparation in Section 7.6) and a blank (CCB made up of 1% NaOH) and are required after every 10 or fewer samples and at the end of the run.

Acceptance Criteria: The measured result for the CCV must be within 10% of the expected value, and the CCB must be less than the reporting limit.

Corrective Action: If either or both the CCV and CCB fail, all samples since the last successful calibration check must be reanalyzed.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≥0.995 correlation coefficient
Initial Cal. Verification (ICV)	Immediately after the calibration	± 10% of the expected value
Initial Cal. Blank (ICB)	Immediately after the calibration	Less than the reporting limit
Continuing Cal. Verif. (CCV)	Prior to / after every 10 injections	± 10% of the expected value
Continuing Cal. Blank (CCB)	Prior to / after every 10 injections	Less than the reporting limit

10.9 Sample Analysis

10.9.1 Following instrument set up and calibration, the sample distillates are analyzed in exactly the same manner as the calibration standards. The routine run sequence is as follows:

Cal 0.00 ppm
Cal 0.01 ppm
Cal 0.02 ppm
Cal 0.05 ppm
Cal 0.10 ppm

Cal 0.20 ppm
Cal 0.40 ppm
Second-source ICV
ICB
High concentration distilled standard (HLCS)
Low concentration distilled standard (LLCS)
LCS
LCSD
Method blank
5 samples (may include MS/SD)
CCV
CCB
10 samples (may include MS/SD)
CCV
CCB
Additional cycles of 10 samples with CCV/CCB
Closing CCV
Closing CCB

10.10 Instrument Shut-Down

10.10.1 Disconnect the reagent lines and put all of them into the DI water, except the buffer line.

10.10.2 The buffer line is put into the bridge water.

10.10.3 Rinse all tubes for at least 5 minutes

10.10.4 Switch the reagent lines to Kleenflow Base and rinse for 5 minutes

10.10.5 Switch the reagent lines to DI water and rinse for 5 min.

10.10.6 Turn off instrument and pump.

10.10.7 Raise platens.

10.10.8 Empty Waste.

10.11 Troubleshooting / Maintenance

10.11.1 Ensure vacuum system is free of leaks by *lightly* applying stopcock grease to all ground glass joints. DO NOT over-grease.

10.11.2 Ensure chiller is on and functioning.

10.11.3 Ensure glassware is free of etching and chips.

10.11.4 Ensure standard solutions are at pH ≥12.

10.11.5 Verify instrument reagent flows are proper and that pump tubing is in good condition.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate SOP, CA-Q-S005, "Calibration Curves" and under the public folder, Arizona Calibration Training.

11.2 Total Cyanide

11.2.1 A linear calibration model is used to relate the cyanide concentration to the absorbance as follows:

$$y = mx + b \qquad \textit{Equation 4}$$

Where:

y	=	Absorbance of cyanide standard at 570 nm.
x	=	Cyanide concentration of standard, mg/L.
m	=	Slope of the fitted straight line.
b	=	y-intercept of the fitted straight line.

11.2.2 The cyanide concentration in an unknown aqueous sample extract is calculated by solving the calibration equation (Equation 1) for concentration (x) and using the measured absorbance of the sample, as follows:

$$x = \frac{y - b}{m} \qquad \textit{Equation 5}$$

Where:

x	=	cyanide concentration in sample (mg/L)
y	=	absorbance of the distillate at 570 nm
m	=	slope of the calibration line
b	=	y-intercept of the calibration line

11.2.3 If an aqueous sample was diluted, use Equation 5 to calculate a final result.

$$C_s = x \times DF \qquad \textit{Equation 6}$$

Where:

C_s	=	Cyanide concentration in the original sample (mg/L).
x	=	Cyanide concentration in sample distillate (mg/L).
DF	=	Dilution factor, if applicable.

11.2.4 The cyanide concentration in an unknown solid sample extract is calculated using the following equation:

$$C_s = \frac{x \times V_t}{W \times D} \times DF \qquad \textit{Equation 7}$$

Where:

C_s = Cyanide concentration in the original sample (mg/L)
x = Extract analyte concentration, mg/L
V_t = Volume of distillate, L (nominal 0.050 L)
W = Weight of sample, kg (nominal 0.001 kg)
D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis
DF = Dilution factor, if applicable.

NOTE: All routine calculations for total cyanide are performed by the instrument data system and the LIMS, provided dilutions and other information have been correctly entered.

11.3 Cyanide Amenable to Chlorination:

$$\text{Amenable Cyanide} = \text{Total CN Result} - \text{Treated Result}$$ *Equation 8*

11.3.1 The "Total CN Result" is the cyanide concentration for the sample aliquot that was distilled without treatment with chlorine. The treated result is the sample portion that was chlorinated and then distilled. The treated result is reported as Nonamenable Cyanide (the cyanide remaining after chlorination) and is calculated in the same manner as Total Cyanide. See Section 11.2 for detailed descriptions of the calculations.

11.3.2 If the chlorinated aliquot shows more cyanide than the unchlorinated aliquot, a corrective action and/or a discussion in the final report is required. Iron-cyanides can cause this to occur. Weak acid dissociable cyanide would be a better method for these types of samples.

11.4 Weak Acid Dissociable Cyanide

The concentration of cyanide measured after the procedure in Section 10.6 is the weak acid dissociable cyanide. See Section 11.2 for detailed descriptions of the calculations.

11.5 Accuracy

ICV, CCV, LCS, HLCS, LLCS:

$$\%\ \text{Recovery} = \frac{\textit{observed concentration}}{\textit{known concentration}} \times 100\%$$ *Equation 9*

MS/MSD

$$\%\ \text{Recovery} = \frac{(\textit{spiked sample conc.}) - (\textit{unspiked sample conc.})}{\textit{spike concentration}} \times 100\%$$ *Equation 10*

11.6 Precision (RPD)

$$\%\ \text{RPD} = \frac{|\,\textit{orig. sample value} - \textit{dup. sample value}\,|}{[(\textit{orig. sample value} + \textit{dup. sample value})/2]} \times 100\%$$ *Equation 11*

11.7 Data Upload

Refer to Work Instruction WI-DV-0068, Cyanide Upload, for upload instructions into TALS.

11.8 Reporting

11.8.1 Reporting units are mg/L for water samples and mg/Kg for solids samples.

11.8.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.8.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.8.4 All associated data are entered or uploaded into the LIMS as required. For soil samples, the total cyanide concentration is calculated by the LIMS using equation 7. If the non-amenable cyanide data are uploaded, the soil concentration is also calculated by the LIMS. If the non-amenable cyanide data are manually entered, the analyst must first calculate the soil concentration from the instrument result using equation 11.2.4. In either case, the amenable cyanide result is calculated by the LIMS using equation 8. If dilutions are performed, the amenable cyanide result in TALS does not reflect an adjusted RL due to the dilution.

Note: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.9 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for an example of the checklist and for more detail on the review process.

12.0 <u>Method Performance</u>

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy No. DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements indicate a greater frequency.

12.1.2 The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of capability (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.1 Four aliquots of the QC check sample or LCS (independent source from the calibration) and a method blank are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample (or LCS) should be equivalent to a mid-level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. The method blank must be less than ½ the RL. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use, has the required experience, and has successfully analyzed initial demonstration samples (see SOP DV-QA-0024 for details).

13.0 Pollution Control

13.1 In general, the quantity of chemicals purchased by TestAmerica Denver is based on expected usage during its shelf life. The volume of reagents and standards prepared for this procedure reflects anticipated usage.

13.2 Source reduction is achieved through the use of midi-distillation followed by an automated colorimetric determination.

13.3 The volume of hazardous waste is minimized through proper segregation and management of the various waste streams generated by this procedure.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Environmental Health and Safety Manual for “Waste Management and Pollution Prevention.”

14.2 The following waste streams have been identified for this method:

14.2.1 Cyanide standardization waste – Aqueous Alkaline (E)

14.2.2 Distilled sample – Aqueous Acidic (F)

14.2.3 Distillate – Aqueous Alkaline (E)

14.2.4 Alpkem process waste – Aqueous Alkaline, contains pyridine (E)

14.2.5 Contents of sampler cups – Aqueous Alkaline (E)

14.2.6 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Standard Methods for the Examination of Water and Wastewater, On-line Edition.

15.1.1 4500-CN^- A-1999. Introduction

15.1.2 4500-CN^- B-1999. Preliminary Treatment of Samples

15.1.3 4500-CN^- C-1999. Total Cyanide after Distillation

15.1.4 4500-CN^- E-1999. Colorimetric Method

15.1.5 4500-CN^- G-1999. Cyanides Amenable to Chlorination after Distillation

15.1.6 4500-CN^- I-1999. Weak Acid Dissociable Cyanide

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.2.1 Method 9012A, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 1, December 1996.

15.2.2 Method 9012B, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 2, August 2002.

15.3 OI Manual (available on instrument computer)

15.4 Department of Defense Quality Systems Manual for Environmental Laboratories, DoD QSM Version 5.0, July 2013.

16.0 Method Modifications:

Item	Method[1]	Modification
1	SM 4500-CN^- SW 9012A SW 9012B	This SOP substitutes bleach for calcium hypochlorite solution.
2	SM 4500-CN^-	There are differences among the referenced methods concerning the sodium hydroxide concentration in working standards: Standard Method 4500-CN E states in Section 4a that working standards are made using a solution containing 1.6 grams per liter of water, which is equal to 0.04 N. Methods 9012A and 9012B state in Section 7.4.1 that calibration standards are prepared using 50 mL of 1.25N sodium hydroxide and diluting to 250 mL, which produces a 0.25 N sodium hydroxide concentration. This procedure uses 0.25 N NaOH to ensure stability of standards. This is the same concentration used in EPA Method 335.4 and SW-846 Method 9012.
3	SM 4500-CN^- SW 9012A SW 9012B	The stock standard is verified if no certificate of analysis is available. Monthly verifications are sufficient to monitor the concentration due to the rate of use of the standard.
4	SM 4500-CN^- SW 9012A SW 9012B	The reflux time for Cyanide Amenable to Chlorination and Total Cyanide has been changed to 1.5 hours versus 1.0 hours to accommodate the reflux time for samples requiring distillation under the Clean Water Act (EPA Method 335.4).
5	SM 4500-CN^-	When the sulfide test is positive for analysis by SM 4500-CN^-, the samples will be treated with cadmium chloride rather than the lead(IV) carbonate listed in SM 4500-CN^- B or lead(II)carbonate listed in SM 4500-CN^- C. This change reduces environmental pollution and provides a standardized sulfide treatment across all cyanide methods.
6	SM 4500-CN^- SW 9012A SW 9012B	SM 4500-CN^- B-1999 states that use of ascorbic acid be used for preservation when residual chlorine is suspected. 40 CFR Part 136.3, Table II states use of reducing agent with none specified. This laboratory uses the ascorbic acid rather than sodium arsenite as sodium arsenite is a hazardous material and the regulation is not explicit regarding which reducing agent to use.
7	SW 9012A SW 9012B	Methods 9012A and 9012B state the amenable cyanide test must be performed under amber light. In this procedure the beakers and watch glasses are wrapped with foil or kept in the dark by alternate means as described in SM 4500-CN^-.
8	SW 9012A SW 9012B	Calibration is verified with an independently prepared check standard (ICV) with every analytical run and a CCV is run after every 10 samples, instead of for every 15 samples. Acceptance criteria used is $\pm$ 10% rather than $\pm$ 15%.
9	SW 9012A SW 9012B	Methods 9012A and 9012B state the use of a 15 mm flow cell. The equipment used in this laboratory utilizes a 10 mm flow cell, which is that specified in SM 4500-CN^-.
10	SW 9012A SW 9012B	Methods 9012A and 9012B utilize the method of standard additions if MS/MSD recoveries exceed acceptance limits. Because MS/MSD results may not have a direct bearing on other samples in the batch, matrix effect is assumed if MS/MSD recoveries exceed acceptance limits when LCS recoveries are

Item	Method[1]	Modification
		acceptable.
11	SM 4500-CN$^-$ SW 9012A SW 9012B	Chloramine-T solution is prepared by adding 2.0 g Chloramine-T to deionized water and diluting to 250 mL. Methods 9012A/B use a proportion of 1.0 g to 250 mL and SM 4500 CN uses 1 g to 100 mL. This laboratory has determined that the more concentrated solution, consistent with the SM 4500 CN provides more consistent coloration of samples.

[1] SM 4500-CN$^-$ refers to approved version 1999 or 2011.

17.0 Attachments

Attachment 1: Example Cyanide Preparation Bench Sheet
Attachment 2: Cyanide Midi-Distillation Apparatus
Attachment 3: Alpkem Manifold Schematic

18.0 Revision History

- Revision 4, dated 9 April 2014
  - Indented sections 7.3.2.9 and 7.3.2.10 as 7.3.2.8.1 and 7.3.2.8.2 to clarify that a new standard is created ONLY if the re-standardized value is <97%
  - Revised section 7.4 to clarify preparation of intermediate standard
  - Revised Section 7.5 to reflect current practice (2 mL changed to 10 mL)
  - Revised table of calibration standards to reflect current practice and match those in SOP DV-WC-0091.
  - Revised Section 7.8.6 to remove filtering of barbituric acid
  - Revised Section 7.9.3 to state solution stored at room temperature
  - Revised Section 7.9.4 to specify solution filtered through glass fiber filter
  - Revised Section 7.12 to reflect current practice
  - Revised Section 7.13 to reflect current practice
  - Added use of plastic container for storage in Section 7.20.3
  - Section 7.24.1: Changed amount and final volume of sulfamic acid prep to match practice. Added storage requirements.
  - Specified aldehyde interferences must be client identified for treatment at lab in Section 7.26.1.
  - Revised Section 7.27 to reflect current practice
  - Changed use of Ottawa sand to Teflon chips for solid matrix for QC samples throughout.
  - Revised Section 9.4.1 to reflect current practice
  - Removed Note from Section 9.4.1 since second source standard is always used for LCS spike.
  - Change acceptance criteria to reflect only HLCS in Section 9.6.2.1.2.
  - Revised Section 10.3.2.1 to record sulfide test results in TALS prep batch
  - Revised Section 10.4.2 from covering the beakers with aluminum to covering with a large plastic tub
  - Dropped reference to Method 335.4 from sample preservation; covered in a separate SOP.
  - Revised Section 10.5.9.2 to use MSA only when specified for a project under DoD QSM 5.0.
  - Revised Section 10.5.14 & 10.5.15 to reflect use of automatic timer.
  - Revised Section 10.5.16 wording for clarity and added note for emphasis.

- Revised Section 10.5.19 to store distillates in sample fridge to distinguish from standard fridge
- Added Trouble Shooting and Maintenance to Section 10
- Revised Method Modification 6 to clarify use of ascorbic acid for all methods in place of sodium arsenite.
- Added criteria for DoD QSM 5.0 throughout.
- Removed reference to SM 4500 CN^- - 1997.

- Revision 3, dated 31 August 2013
  - Added TALS reagent module IDs for reagents and standards in section 7.0
  - Revised concentration of Intermediate Standard I to reflect current practice
  - Added note to section 7.9.2 to address potential need for addition of more Brij-35 solution for optimal instrument performance
  - Increased amount of Chloramine-T in section 7.10 to 2.0 g for improved performance
  - Revised sample preservation pH to comply with EPA 2012 MUR for NPDES compliance samples (Methods 334.1 and SM 4500 CN-)
  - Updated MB acceptance criteria to reflect current practice , Sections 9.2 & 9.3
  - Corrected units in section 11.2.4
  - Added new section 11.7 on data upload instructions
  - Revised Section 11.8.4 for clarification in handling data required for measuring the amenable cyanide concentration
  - Added item 11 to Method Modification table
  - Added requirements of 12 QC elements specified in EPA 2012 MUR to 40 CFR Part 136 throughout

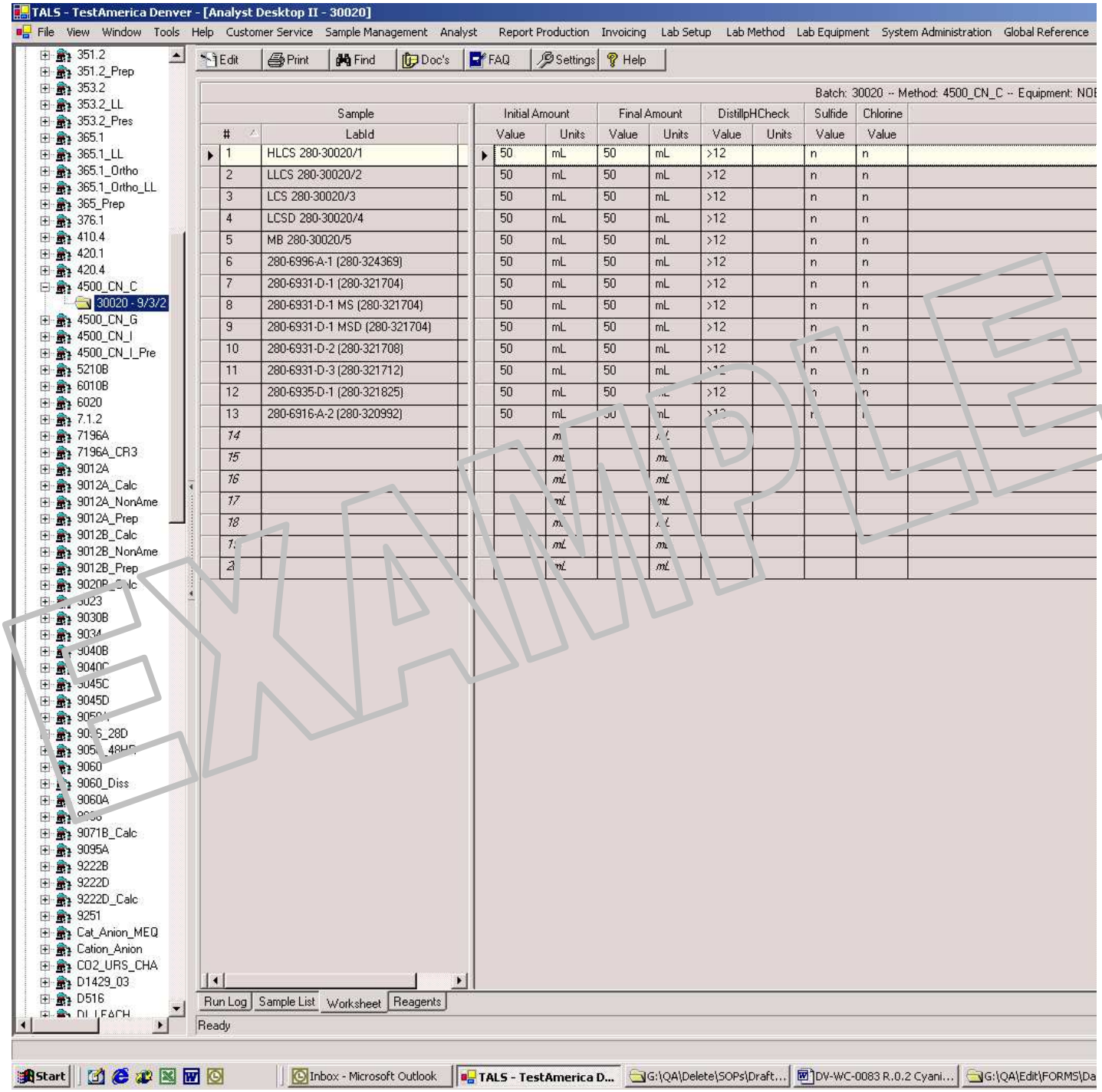
- Revision 2, dated 31 August 2012
  - Combined SOP DV-WC-0082 with this SOP and retired SOP DV-WC-0082 highlighting details that are specific to the respective source methods
  - Added Sections 2.3 and 2.4
  - Renumbered and reordered ICAL standards table to be consistent with numbering used at the bench (Section 7.5.2)
  - Updated Section 9 and 10 to clarify which batch QC samples and their frequency are performed for each type of cyanide determined.
  - Revised order in Section 11, added equation for calculation of concentration in solid samples
  - Removed reference to Method 9013
  - Added references for SM 4500-CN^- - 1999 (per 2012 MUR)
  - Updated method modifications table for Methods 9012A and 9012B (#1,3,4) and added modifications 6-10
  - Source method review

- Revision 1.1, dated 18 June 2012
  - Updated section 9.1 for consistency with other SOPs.
  - Updated volumes for spiking LCS/MS/MSD/HLCS and LLCS and concentration of standard used to reduce volumes used.
  - Added section 10.1 and 10.2 for consistency with other SOPs.
  - Revised statement regarding frequency of standard verification in Section 16 consistent with procedure.
  - Corrected typographical errors and section references throughout.

- Revision 1, dated 04 April 2012
  - Source method review
  - Expanded Section 1 to include discussion of differences between Cyanide Amenable to Chlorination and Weak Acid Dissociable Cyanide (WAD). Added WAD and Free Cyanide to list of analytes and listed RLs.
  - Added definitions of the forms of cyanide (Section 3)
  - Clarified when standardization of stock standard is required.
  - Changed “Standards Log” to “Reagent Module” in the LIMS to describe current practice.
  - Added procedural steps for preparation of soil samples and associated QC samples (Section 9 and 10).
  - Added SM requirement to spike LCS and MS/MSD with second source standard
  - Added requirement to protect samples from light during chlorination step
  - Added equation for linear regression and use of that equation for calculation of sample concentration (Section 10 and 11).
  - Reordered Section 11.
  - Added method blank requirement from Standard Methods to IDOC procedure (Section 12.3)
  - Replaced Attachment 1 with example of worksheet in TALS
  - Removed Attachment 2 and renumbered existing attachments
  - Replaced diagram of distillation apparatus with midi-distillation apparatus more comparable to equipment in use.
  - Formatting and grammatical changes throughout
- Revision 0.2, dated 31 March 2011
  - Annual Technical Review
  - Removed the Note from Section 10.6.2 – distillation of curve
  - Corrected grammar and formatting errors
  - Updated Attachment 1 & 2
  - Added Section 11.1 referencing corporate SOP CA-Q-S005, “Calibration Curves”.
- Revision 0.1, dated 13 March 2010
  - Annual Review
  - Added section 6.3
- Revision 0, dated 27 February 2009

Attachment 1.

Example Cyanide Preparation Bench Sheet



EXAMPLE

Attachment 2.

Cyanide Distillation Apparatus

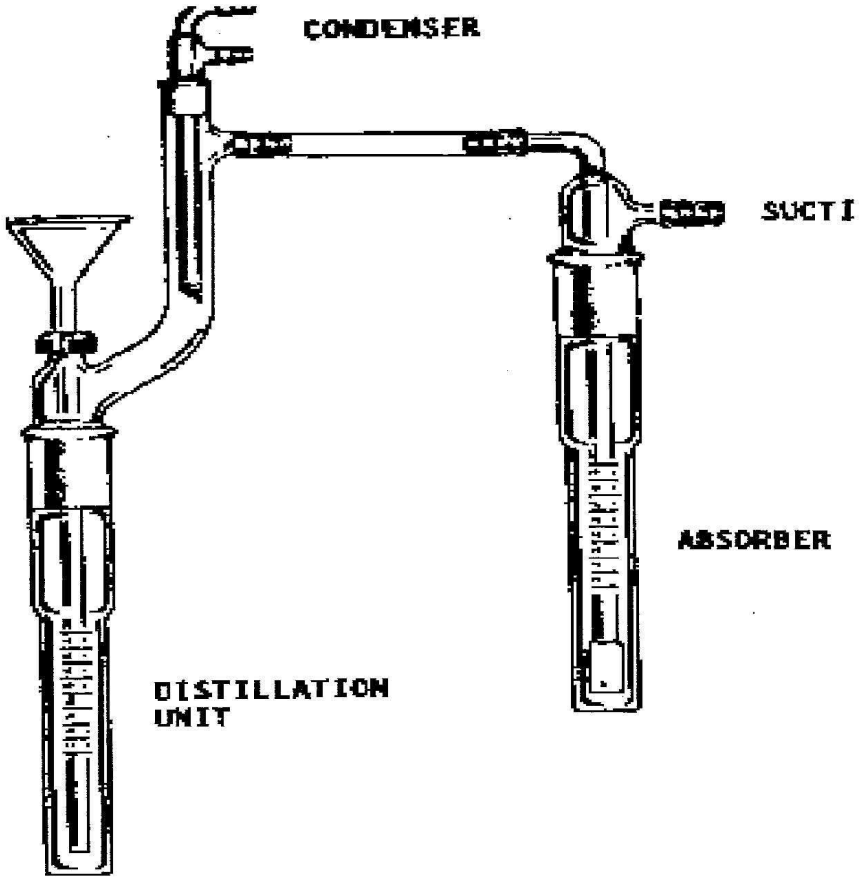
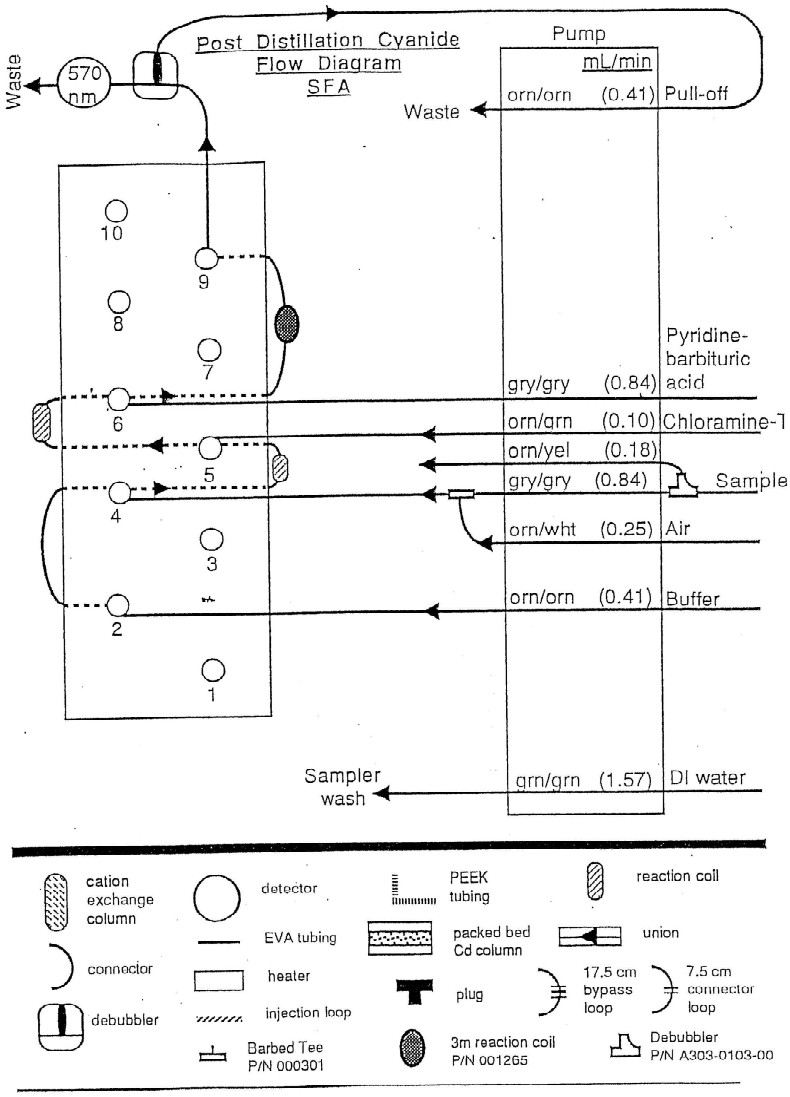


FIGURE 1. MIDI DISTILLATION APPARATUS

335.4-15

Attachment 3.

Alpkem Manifold Schematic



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Title: Organochlorine Pesticides by Gas Chromatography Based on SW-846 Methods 8000B/8000C, 8081A/8081B, Method 608 and Compendium Methods TO-4A, and TO-10A

[Methods 8000B, 8000C, 8081A, 8081B, 608, TO-4A, TO-10A]

Approvals (Signature/Date):

Victoria Nihart 10/16/14	Joe Schairer 7/23/14
Victoria Nihart — Date	Joe Schairer — Date
Technical Manager	Health & Safety Manager / Coordinator
Lisa Stafford 7/22/2014	Robert Hrabak 7/24/14
Lisa Stafford — Date	Robert Hrabak — Date
Interim Quality Assurance Manager	Laboratory Manager

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1. SCOPE AND APPLICATION

This SOP describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). This SOP is applicable to extracts derived from any matrix which is prepared according to the appropriate sample extraction SOP (WS-OP-0002).

This procedure is also applicable to the determination of organochlorine pesticides in municipal and industrial discharges under 40 CFR 136.1.

Table 1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

1.1. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. Aqueous samples are prepared for analysis using continuous or separatory funnel liquid-liquid extraction or solid phase extraction, and semivolatile analytes in solid samples are prepared using sonication, Soxhlet extraction, or microwave assisted extraction.

After the initial preparation step, the sample is introduced to the chromatograph and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. The two injections following a high level sample should be evaluated for possible carryover. A solvent blank may be used following high level samples to monitor for carryover.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP WS-OP-0002.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, as outlined in the preparation SOP WS-OP-0002.

4.5. Aroclor mixtures will interfere with pesticide analysis. If 8082 results indicate the presence of Aroclors, raising of reporting limits may be required due to matrix interferences.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The autovials used on the instrument contain a small amount of solvent after the analysis is completed, which can evaporate into the air through the puncture in the septa. These autovials may be collected in an open tripour or similar cup at the instrument while the analyst is removing them from the tray, but once that is complete the vials must be transferred to a waste container in a fume hood. They cannot be left out on the bench by the instrument.

5.1.2. Ensure that the outlet lines from the instrument are secured and lead into either a fume hood or inlet point to the fume hood exhaust system.

5.1.3. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.1.4. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing the EH&S coordinator shall be immediately notified and a letter sent to the

NRC or local state agency.

5.1.5. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when working with open sample extracts, spikes, surrogates or solvents. Latex or vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should only be used when working with closed extract vials.

5.1.6. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.7. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2. An analytical system complete with a gas or liquid chromatograph is required. A data system capable of measuring peak area and/or height is required.

6.2.1. Gas Chromatograph - Agilent model 6890N or equivalent.

6.2.2. Autosampler – Agilent model 76373, 7683B or equivalent.

6.2.3. Operating System – Agilent NSD Chem Station D.02-00-275 or equivalent.

6.2.4. Software - Chrom Peak Review, Version 2.1 2012.1129 or equivalent.

6.2.5. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.3. A ^{63}Ni electron capture detector is required.

6.4. Analytical columns: Stx-CLPesticides 30m x 0.32 mm x 0.50µm and Stx-CLPesticides II 30m x 0.32 mm x 0.25µm, or equivalent.

6.5. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Gases for carrier and make-up: Hydrogen, Helium, and Nitrogen.

7.2. Solvent for dilutions (if needed) and syringe cleaning: Hexane, pesticide grade.

7.3. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If a vendor supplied standard has an earlier expiration date then that date is used.

7.4. Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standard solutions are stored at 0 - 6ºC or at manufacturer's recommended storage temperature. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using. Stock standard solutions must be replaced after one year.

7.5. Column Degradation Evaluation Mix

A mid level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table 5 for details of the column degradation evaluation mix.

7.6. Calibration Standards

Calibration standards are prepared as dilutions of the stock standards. Calibration solutions must be refrigerated at 0 - 6ºC and protected from light. The standards must be replaced at least every six months or sooner if comparisons with check standards indicates a problem. Refer to Table 4 for details of calibration standards.

7.7. Initial Calibration Verification (ICV)

The ICV is a second source standard prepared at a concentration approximately mid-way between the highest and lowest calibration standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Extracts must be refrigerated at 0 - 6 ºC and analyzed within 40 days of the end of the extraction.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. The minimum initial demonstration required is analysis of a single point calibration.

9.2. Quality Control Batch

9.2.1. The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch may contain multiple MS/MSDs.

9.2.2. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible. QC samples should be analyzed with the field samples; however, field samples may be split between instruments or sequences as needed. Refer to the QC Program document (WS-PQA-003) for further details of the batch definition.

9.2.3. If a QC batch is analyzed on more than one instrument, at the minimum an instrument blank must be analyzed on the second instrument to demonstrate that the system is free of interferences.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). Refer to policy WS-PQA-003 for more details.

9.3.1. These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless there is no response after dilution.

9.3.2. All surrogate, LCS, and MS recoveries must be entered into LIMS or other database so that accurate historical control limits can be generated.

9.3.3. Refer to the QC Program document (WS-PQA-003) for further details of control limits.

9.4. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented using the NCM module in TALS.

9.5. Surrogates

Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. Both surrogates used in this analysis must meet the recovery criteria. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Reprepare and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem. Repreparation is not necessary if there is obvious chromatographic interference or other evidence of matrix impacts.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that

poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.5.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.3). The column used to confirm positive results must meet all quality control criteria (retention times and continuing calibration verification). Primary column should be reported for surrogates for DOD projects, rather than higher value.

9.5.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then re-preparation or flagging of the data is required.

9.5.3. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.6. Method Blanks

One method blank must be processed with each preparation batch. The method blank consists of reagent water for aqueous samples, and sodium sulfate for solid samples (refer to the appropriate preparation SOP for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher. Certain programs may require a more stringent evaluation of the method blank. For instance, DOD requires that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.

- Re-preparation and reanalysis of any samples with reportable concentrations of analytes less that 10 times the value found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. This must be documented in the NCM program.
- If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.6.1. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.7. Laboratory Control Samples (LCS)

9.7.1. For each batch of samples, analyze a LCS. At a minimum, the LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes if so required by the client or program governing the samples, such as DOD. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. For method 608, an LCS is required as 10% of all samples analyzed.

9.7.2. If dual column analysis is used the choice of which result to report is described in Section 12. The primary column should be reported for QC for DOD, rather than higher value.

9.7.3. LCS compound lists are included in the appendices.

9.7.4. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client. DOD allows one sporadic marginal exceedance for LCS spikes with 11-30 analytes. See WS-PQA-021 and WS-PQA-003 for further explanation for sporadic marginal exceedances.

9.7.5. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:

- Check calculations,
- Check instrument performance,
- Reanalyze the LCS, and if still outside of control limits,
- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.7.6. The analyst should evaluate the anomalous analyte recovery for possible trends, as described in the QC Program document (WS-PQA-003). Control charts will be used to track trends and sporadic marginal exceedances.

9.7.7. Data may be reported with an anomaly in the following cases:

- The LCS recoveries are high and the analyte of concern is not detected in field samples,
- All target requested analytes are within control, but other LCS compounds are out of control.

9.7.8. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

9.7.9. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.7.10. If the batch is re-extracted past sample holding time, both sets of results should be reported, flagged and narrated.

9.7.11. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits, as described in the QC Program document (WS-PQA-003). Method 608 requires a matrix spike on at least 10% of the samples from each sample site being monitored.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed.
- The MS/MSD must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report is described in Section 12. The primary column should be reported for QC for DOD, rather than higher value.

9.8.2. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. File an NCM module in TALS stating that the 4X rule was applied. This NCM must be included in the final report.

9.8.3. For Method 608, the spike concentration for each parameter is found in Table 7. Compare the percent recovery (P) for each parameter with the corresponding

QC acceptance criteria. If the spike concentration was lower than the concentration in Table 7, the acceptance criteria must still be met or use the optional acceptance criteria in Table 8. .

9.8.3.1. If the recovery (P) is outside the acceptance criteria in Table 7 a QC check standard (QCCS / LCS) is analyzed and the percent recovery (Ps) is compared to Table 7. Analytes that fail in the matrix spikes and QCCS / LCS) are out of control and can not be reported for regulatory compliance purposes.

9.9. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.11. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003).

10. CALIBRATION

10.1. Column Degradation Evaluation

The column evaluation mix or performance evaluation mix (PEM) must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see Section 12.8.3) and each shown to be ≤15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of a capillary column.
- Replacing the GC column.

10.2. Initial Calibration

External calibration is used. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the Table 4.

10.2.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new (uncalibrated) columns, or replacing the ECD detector. A new calibration is not required after performing column maintenance, replacing the septum or syringe, or performing other minor maintenance.

10.2.2. With the exception of instances detailed in Policy CA-P-T-002, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points.

10.2.3. The initial calibration consists of an injection of the column degradation evaluation mix, a minimum of 5 levels of the individual pesticide (IND AB) standard (Table 4), the ICV for INDAB, and a mid-level toxaphene standard, analyzed in that order. If necessary, the additional compound mixture ("ADD" mix) and technical chlordane may also be injected.

10.2.4. The surrogate calibration curve is calculated from the INDAB mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.5. Prior to accepting an initial calibration curve, it is highly recommended to recalculate the calibration levels using the new calibration factors. Calculate the % difference for every analyte, at each level of calibration. These values should be compared to the criteria below and justified if not met.

10.2.5.1. For methods 608, 8081A, TO-4A and TO-10A, the criteria is ± 15% difference for each analyte.

10.2.5.2. For method 8081B, the criteria is ± 20% difference for each analyte

10.2.6. For multi-component pesticides:

10.2.6.1. To quantitate multi-component analytes, select 4-10 major peaks in the analyte pattern. Calculate the response using the total area of these peaks. Use this response in equation 1 to calculate the response factor.

10.2.6.2. For multi-component analytes, the mid level standard must be analyzed as part of the initial calibration. This single point calibration is used to identify the presence of multi-component analytes.

10.2.6.3. If a multi-component pesticide is identified as present in a sample, the sample is reanalyzed following a full multi-point initial calibration for the pesticide.

10.2.6.4. The analyst may analyze a full multi-point calibration for any of the multi-component analytes at the same time as the calibration for the IND AB calibration if prior history of expected samples indicates a need, or at client request.

10.2.6.5. Refer also to white paper CA-Q-QM-003, Technical Guidance on Reporting Multi-component Samples.

10.2.7. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak response to the mass or concentration injected may be used to prepare a calibration curve. Peak response is measured as the area of the peak.

Equation 1

$$\text{Calib Factor (CF)} = \frac{\text{Sum of peak areas}}{\text{Concentration of Solution (ng/mL)}}$$

10.3. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor is the preferred fit, and may be used if the % RSD of the response factor or calibration factor of each analyte is ≤ 20% (< 10% for Method 608). If an analyte exceeds the criteria, then a regression line (linear or curved) should be attempted. If the regression lines do not meet criteria (coefficient of determination, $r^2 > 0.99$, or correlation coefficient, $r \geq 0.99$), evaluate the calibration, then perform any required instrument maintenance prior to repeating the initial calibration process

10.3.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.3.2. Average response factor

The average response factor may be used for an analyte if the percent relative standard deviation (%RSD) of the response factors for a given analyte is ≤ 20%. Method 608 requires the %RSD to be <10% for the average response factor. The equation for average response factor is:

Equation 2

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.3.3. Linear regression

The linear fit uses the following function:

Equation 3

$$y = ax + b \quad \textbf{or} \quad x = \frac{(y-b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.3.4. Quadratic curve

The quadratic curve uses the following function:

Equation 4

$$y = ax^2 + bx + c$$

Where: y = Instrument response
x = Concentration
a = Curvature
b = Slope
c = Intercept

10.4. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve fit (linear or otherwise) is used, the intercept of the curve at zero response must be less than ± ½ the reporting limit for the analyte.
- For the linear case, the correlation coefficient (r) must be greater than or equal to 0.990. For the quadratic case the coefficient of determination (r^2) may be used, and must be greater or equal to 0.990.

10.5. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If calibrations are required, calibration data quality objectives may need to be established with the client.

10.6. Initial Calibration Verification (ICV): A second source standard must be analyzed with the initial calibration curve. Each compound of the second source calibration must be within ± 20% (Method 8081B) or ± 15% (Methods 8081A, TO-4A, and TO-10A) of its expected value when calculated against the initial calibration. Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards
- Re-calibrate as needed

10.7. Continuing Calibration Verification (CCV)

The calibration verification sequence must be initiated within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed, or following every 20 samples, whichever is more frequent. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a CCV. Mid level calibration standards are used for the 12 hour calibration. The working calibration curve or calibration factor for Method 608 must be verified on each working day prior to sample analysis.

10.7.1. At a minimum, the 12 hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multicomponent analytes. (For example, PEM, INDAB, TOX, CHLOR standards would all be analyzed if the analytes requested were the single-eluting pesticides plus toxaphene and technical chlordane.)

10.7.2. The retention time windows for any analytes included in the 12 hour calibration are updated.

10.7.3. At a minimum, the working calibration curve or RF must be verified by the analysis of a mid-level calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. Some programs, such as DOD, may require more frequent verification, or base the frequency on the number of samples instead of a time period. DOD requires a CCV for every ten injections of field samples. The center of each retention time window is updated with each 12 hour calibration. The initial calibration verification standard used at the start of the analysis sequence must be a different concentration than the mid-level calibration standard used for subsequent calibration verifications. Recommended concentrations are given in the method appendices.

10.7.4. It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed,

requirements are the same as the 12 hour calibration with the exception that retention times are not updated.

10.7.5. Any individual compounds with %D ≤ 20% (Method 8081B) or ≤ 15% (Methods 8081A, Method 608, TO-4A, and TO-10A) meets the calibration criteria.

10.7.6. It is not necessary to run a daily continuing calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration, i.e., the calibration curve and second source standard (initial calibration verification).

10.7.7. Samples must be bracketed by calibration verification standards that meet the criteria listed above.

10.7.8. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria and the preceding samples show no evidence of similar failure, the preceding samples have been successfully bracketed and no further action is required. If this CCV fails or adjustments to the instrument are performed before the repeat CCV then the preceding samples have not been successfully bracketed but analysis may continue.

10.7.9. It is not allowed to analyze repeat CCVs on unattended runs.

10.7.10. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.7.11. % Difference calculation

% Difference is calculated as follows:

Equation 5

$$\%Difference = \frac{\left| X - \overline{X} \right|}{X} \times 100\%$$

Where X = Calibration or response factor of the CCV

$\overline{X}$ = Average Calibration or response factor.

10.7.12. % Drift calculation

% Drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for % drift are the same as for % difference

Equation 6

$$\%Drift = \frac{|CalculatedConcentration - TheoreticalConcentration|}{TheoreticalConcentration} \times 100\%$$

10.7.13. Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 20% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than ± 20%, a new calibration curve must be prepared.

10.7.13.1. Any samples injected after the last good continuing calibration standard must be reinjected, unless one of the following conditions is satisfied:

10.7.13.2. If the CCV response is elevated and the preceding samples are non-detect for all analytes of concern, an NCM may be filed and the data reported.

10.7.13.3. If the samples have already been analyzed twice, and it is apparent that matrix effects are causing the out of control event, an NCM may be filed and the data reported. Client consultation or dilution of samples may be necessary.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Extraction and cleanup procedures are referenced in SOP WS-OP-0002.

11.3. Refer to Table 3 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table 1 on both columns.

11.4. Allow extracts to warm to ambient temperature before injection. Extracts are introduced by direct injection. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. The suggested analytical sequence is given in Table 7.

11.5.1. The daily calibration includes analysis of standards containing all single response analytes, and updating the retention time windows.

11.5.2. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

11.5.3. Solvent blanks may be analyzed immediately following CCVs or suspect samples at any point during the run, however, they may not be routinely used to "clean out the instrument" immediately prior to CCVs.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte. For multi-component analytes (e.g., toxaphene) use the retention time of major peaks. The retention time window is defined as plus or minus three times the standard deviation of the retention times of each analyte.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid point of the initial calibration and each 12 hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than ± 0.03 minutes, use ± 0.03 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by the 12 hour calibration.

11.7. Daily Retention Time Windows

The center of the retention time windows determined in Section 11.6 is adjusted to the retention time of each analyte as determined in the 12 hour calibration standards. Multi-component analytes are only updated when used for quantitation. The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration, but not for other bracketing calibration verification standards.

11.8. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP WS-OP-0013 for determination of percent moisture.

11.9. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Identification

Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Confirmation is required on a second column except for multi-component analytes. Client specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Second column confirmation of multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (for DOD, compare to the MDL).

12.2. Identification of Multi-component Analytes

Retention time windows are also used for identification of multi-component analytes, but the “fingerprint” produced by major peaks of those compounds in the standard is

used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Dual channel quantitation

Certain programs, such as DOD, may have differing requirement for the reporting data from dual channel analyses. Refer to the DOD QSM policy (WS-PQA-021) for DOD requirements, or the client QAPPs. DOD QSM projects require approach B below. The column used to confirm positive results must meet all quality control criteria (retention times and continuing calibration verification). For further information regarding dual channel quantitation, refer also to the policy CA-T-P-003, "Dual Column Confirmation".

For confirmed results, two approaches are available to the analyst:

A) The primary column approach

B) The better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.3.1. Primary column approach:

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true:

- There is obvious chromatographic interference on the primary column.
- The result on the secondary column is 40% greater than the result on the primary column, and there is no reason apparent on the chromatogram for rejecting the higher value (i.e., faulty integration, negative dips affecting the baseline, etc.)
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for reanalysis.) In this instance, only ND (not requiring deconfirmation) or spike results may be reported.

LCS, surrogates, and MS/MSD are reported from the primary column unless a standard failure dictates reporting a given analyte from the secondary column. This must be noted in an anomaly.

12.3.2. Better result approach

The higher of the two results is normally reported unless obvious chromatographic interference is noted.

LCS, surrogates, and MS/MSD are reported from the primary column unless a standard failure dictates reporting a given analyte from the secondary column. This must be noted in an anomaly. All corrective action for QC is based from the primary column, even if the secondary column passes QC criteria.

12.3.3. If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect, and the results are qualified. If no anomalies, such as overlapping peaks, are noted, report the higher result, or both results. RPD is calculated using the following formula:

Equation 7

$$RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\%$$

Where R=Result

12.4. Quantitation of Multi-component Analytes

Use 4-10 major peaks for quantitation as described in Section 10.2.5, initial calibration of multi-component analytes.

12.4.1. Multi-response Analytes (e.g., toxaphene or technical chlordane)

12.4.1.1. For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.4.1.2. If required by the client or program, second column confirmation of these analytes may be performed.

12.4.2. The experience of the analyst should weigh in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.5. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix or screening results.

12.6. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.6.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination.

12.6.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request. Projects for DOD QSM require reporting of both dilutions.

12.7. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in a nonconformance memo.

12.8. Calculations

12.8.1. Aqueous samples

Equation 8

$$\text{Concentration (ug/L)} = \frac{A \times D \times FV}{V \times CF}$$

Where:
A = Response for the analyte in the sample
D = Dilution factor
FV = Final extract volume (mL)
V = Volume of sample extracted
CF = Calibration factor, area /(ng/mL), Section 10.1

12.8.2. Non-aqueous Samples

Equation 9

$$\text{Concentration (ug/kg)} = \frac{A \times D \times FV}{CF \times W \times M}$$

Where:
A = Response for the analyte in the sample
D = Dilution actor
FV = Final extract volume (mL)
W = Mass of sample extracted (g)
CF = Calibration factor, area /(ng/mL), Section 10.1

M = Percent Solids, [(100 - %Moisture)/100]

12.8.3. Calculation of Column Degradation/% Breakdown (%B)

Equation 10

$$\%B_{DDT} = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

Where:
$\%B_{DDT}$ = % Breakdown for 4,4'-DDT
A_{DDD} = Area of 4,4'-DDD (if present)
A_{DDE} = Area of 4,4'-DDE (if present)
A_{DDT} = Area of 4,4'-DDT

Equation 11

$$\%B_{Endrin} = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{Endrin}} \times 100$$

Where:
$\%B_{Endrin}$ = % Breakdown for endrin
A_{EK} = Area of endrin ketone (if present)
A_{EA} = Area of endrin aldehyde (if present)
A_{Endrin} = Area of endrin

12.8.4. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

Equation 12

$$\%\ \text{Recovery} = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. 1-2 milliliter autovials containing small amounts of solvent extract. These are collected at the instrument and immediately transferred to a satellite collection container in a fume hood. When full or after no more than one year, these satellite collection containers are transferred to the waste disposal area for disposal.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Method 8000B

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Method 8000C, Revision 3, March 2003.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, Method 8081A, Revision 1, December 1996, Organochlorine Pesticides by Gas Chromatography

16.4. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update IV, Method 8081B, Revision 2, February 2007, Organochlorine Pesticides by Gas Chromatography

16.5. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-4A, January 1999, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)

16.6. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-10A, January 1999, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)

16.7. 40 CFR Appendix A Part 136 Method 608 April 8, 2014.

17. METHOD MODIFICATIONS

17.1. Modifications from Method 8000B

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit.

17.2. Modifications from Reference Methods TO-4A and TO-10A

17.2.1. A minimum 5-point calibration is performed for all single component pesticides. A single point is performed for Technical Chlordane and Toxaphene. An average response factor is determined for each analyte.

17.2.2. The calibration is verified daily or every 12 hours with the mid-point standard, rather than every 10 samples. This is consistent with SW-846 methods.

17.2.3. The RT windows are set using the mid-point standard and are updated daily or every 12 hours from the mid-point standard.

17.2.4. Sample analysis is performed using a RTx-CLPesticides and RTx-

CLPesticides2, or other appropriate GC columns.

17.2.5. Instrument operating conditions will be determined on an instrument and column specific basis.

17.2.6. Method Blanks are deemed acceptable if no target analytes are present at or above the reporting limits or >10% of the measured concentration in the associated samples.

17.2.7. Sample results are not corrected for extraction efficiency, as determined by surrogate recovery.

18. ATTACHMENTS

18.1. Table 1- Standard Analyte List and Reporting Limit

18.2. Table 2 – Initial and Final Volumes

18.3. Table 3 – Parameter and Recommended Conditions

18.4. Table 4 – Calibration Levels

18.5. Table 5 – Column Degradation Evaluation Mix

18.6. Table 6 - LCS/Matrix Spike and Surrogate Spike Levels

18.7. Table 7 – QC Acceptance Criteria – Method 608

18.8. Table 8 – Method Accuracy and Precision as Functions of Concentrations – Method 608

18.9. Table 9 – Summary of Method 608 QC Requirements.

18.10. Table 10 – Suggested Analytical Sequence

18.11. Table 11 – Recommended Maintenance for Method 8081 Instruments

19. REVISION HISTORY

19.1. WS-GC-0001, Revision 5.1, Effective 06/27/2014

19.1.1. Table 2, changed RVE to LVI in column 1 following water by reduced volume extraction and added water volume from 125mL to 250mL.

19.1.2. Table 6, added the sample reduced volume from 125mL to 250mL.

19.1.3. Editorial changes.

19.2. WS-GC-0001, Revision 5, Effective 04/18/2014

19.2.1. Changed title to include Organochlorine Pesticides by Gas Chromatography

19.2.2. Section 1, inserted, "This procedure is also applicable to the determination of organochlorine pesticides in municipal and industrial discharges under 40 CFR 136.1."

19.2.3. Section 9.7, appended, "For method 608, an LCS is required as 10% of all samples analyzed."

19.2.4. Section 9.8, inserted, "Method 608 requires a matrix spike on at least 10% of the samples from each sample site being monitored."

19.2.5. Added Sections 9.8.3 and 9.8.3.1 regarding the criteria to be evaluated for Method 608 MS/MSD.

19.2.6. Language inserted for the spike concentration and acceptance criteria for the QC check standard (QCCS) in Table 7.

19.2.7. Section 10.2.5.1 inserted, "Method 608".

19.2.8. Section 10.3, second sentence, appended, "(< 10% for Method 608)".

19.2.9. Section 10.3.2, inserted, "Method 608 requires the %RSD to be <10% for the average response factor."

19.2.10. Section 10.7 appended, "Method 608 must be verified on each working day prior to sample analysis."

19.2.11. Section 10.7.5, inserted, "Method 608".

19.2.12. Section 16, added reference, "40 CFR Appendix A Part 136 Method 608 April 8, 2014."

19.2.13. Inserted Tables 7, 8, and 9

19.3. WS-GC-0001, Revision 4.6, Effective 09/06/2013

19.3.1. Section 2, paragraph 1, added "or microwave assisted extraction." to the last sentence.

19.3.2. Table 1: Inserted the word "Minimum" into the title.

19.3.3. Table 1: Changed reporting limits for 4,4'-DDE, 4,4'-DDD, 4,4'-DDT, Dieldrin, Endosulfan II, Endosulfan Sulfate, Endrin, Endrin Aldehyde, and

Endrin Ketone from 1.0 to 0.5 ug/L for waters.

19.3.4. Table 1: Changed reporting limits for Methoxychlor from 2.0 to 0.5 ug/L for waters.

19.3.5. Table 1: Changed the reporting limit for Isodrin from 1.0 to 0.2 ug/L for waters.

19.3.6. Table 1: Changed the reporting limit for Technical Chlordane from 5.0 to 0.5 ug/L for waters.

19.3.7. Table 4: Corrected the units to ug/L.

19.3.8. Table 4: Inserted new level 1 values for Isodrin (0.5), Mirex (0.5), Diallate (25), Chlorobenzilate (25), 2,4'-DDT (2.5), 2,4'-DDD (2.5), and 2,4'-DDE (2.5), and shifted the existing levels to be levels 2 through 7.

19.3.9. Table 4: Inserted new level 1 value for Technical Chlordane (12.5) and shifted the existing levels to be levels 2 through 7.

19.4. WS-GC-0001, Revision 4.5, Effective 06/18/2013

19.4.1. Updated SOP to reflect change to TALS system.

19.4.2. Updated to include Method 8000C/8081B criteria. Specific sections changed noted below.

19.4.3. Removed references to TO-4 and TO-10, as these are outdated versions.

19.4.4. Section 9.51, added "The column used to confirm positive results must meet all quality control criteria (retention times and continuing calibration verification)." removed option to use in-control column.

19.4.5. Inserted Section 10.2.5 regarding calculation ICAL standards with the generated calibration factors and evaluating drift with respect to ICAL.

19.4.6. Sections 10.3 and 10.4, changed the correlation (r) value to 0.990 from 0.995, to match the source method.

19.4.7. Updated Section 10.6 to have criteria for both Method 8081A and 8081B.

19.4.8. Updated Section 10.7.5 to have criteria for both Method 8081A and 8081B.

19.4.9. Changed the Table 1 footer to Table 2.

19.4.10. Updated Table 3 to reflect current laboratory practices.

19.4.11. Editorial changes.

19.5. WS-GC-0001, Revision 4.4, Effective 12/28/2012

19.5.1. Updated software to Chrom Peak Review.

19.5.2. Updated oven temperature profile.

19.5.3. Editorial changes.

19.6. WS-GC-0001, Revision 4.3, Effective 10/21/2011

19.6.1. Inserted Table 7 – Recommended Maintenance for Method 8081A Instruments.

Table 1
Standard Analyte List and Minimum Reporting Limits

Compound	Reporting Limit			
	Water µg/L	Soil µg/kg	Waste µg/kg	Air µg/sample
Standard Analyte List				
Aldrin	0.05	1.7	50	0.15
α-BHC	0.05	1.7	50	0.15
β-BHC	0.05	1.7	50	0.15
δ-BHC	0.05	1.7	50	0.15
γ-BHC (Lindane)	0.05	1.7	50	0.15
α-Chlordane	0.05	1.7	50	0.15
γ-Chlordane	0.05	1.7	50	0.15
4,4'-DDD	0.05	3.4	50	0.30
4,4'-DDE	0.05	3.4	50	0.30
4,4'-DDT	0.05	3.4	50	0.30
Dieldrin	0.05	3.4	50	0.30
Endosulfan I	0.05	1.7	50	0.15
Endosulfan II	0.05	3.4	50	0.30
Endosulfan Sulfate	0.05	3.4	50	0.30
Endrin	0.05	3.4	50	0.30
Endrin Aldehyde	0.05	3.4	50	0.30
Endrin Ketone	0.05	3.4	100	0.30
Heptachlor	0.05	1.7	50	0.15
Heptachlor Epoxide	0.05	1.7	50	0.15
Methoxychlor	0.05	3.4	100	1.5
Toxaphene	2.0	67	2000	6.0
APPENDIX IX and other Add-Ons				
Mirex	0.1	1.7	50	--
Diallate	1.0	35	1000	--
Isodrin	1.0	35	100	--
Chlorobenzillate	1.0	35	100	--
Chlordane (technical)	5.0	17	500	--
2,4’-DDD	0.1	3.4	--	--
2,4’-DDE	0.1	3.4	--	--
2,4’-DDT	0.1	3.4	--	--

Table 2 Initial and Final Volumes			
Matrix	**Sample Volume**	**Final Extract Volume**	**Initial Extract (Clean and submitted to GC group)**
Water	1000 mL	10 mL	2 mL
Water by Reduced Volume Extraction (LVI)	250 mL	5 mL	2 mL
	125 mL	5 mL	2 mL
Low Level Soil/Solid	30 g	10 mL	3 mL
Low Level Soil/Solid by EPA 3586 (MAE)	15 g	5 mL	2 mL
High Level Solid/Waste/Oil	1 g	10 mL	2 mL
Air (trapped in PUF or similar)	1 Sample	10 mL	2 mL

Table 3	
Parameter	**Recommended Conditions**
Injection port	200°C, 70.0 psi for 0.5 min in pulsed splitless mode
Detector temp	300°C
Temperature program	120°C for 0 minute - 19°C /minutes to 300°C, hold 1.2 minutes - 30°C /minutes to 315°C, hold 1.0 minute
Flow program	2.0 mL/min
Column 1	Stx-CLPesticides-1 30m x 0.32mm x 0.5µm
Column 2	Stx-CLPesticides-2 30m x 0.32mm x 0.25µm
Injection	2µL
Carrier gas	Hydrogen
Make up gas	Nitrogen
Y splitter	Restek/Agilent/Supelco

Table 4								
Suggested Calibration Levels (µg/L)								
Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
alpha-BHC	1.0	2.0	5.0	10	20	50	100	200
gamma-BHC (Lindane)	1.0	2.0	5.0	10	20	50	100	200
beta-BJC	1.0	2.0	5.0	10	20	50	100	200
delta-BHC	1.0	2.0	5.0	10	20	50	100	200
Heptachlor	1.0	2.0	5.0	10	20	50	100	200
Aldrin	1.0	2.0	5.0	10	20	50	100	200
Heptachlor Epoxide	1.0	2.0	5.0	10	20	50	100	200
gamma-Chlordane	1.0	2.0	5.0	10	20	50	100	200
alpha-Chlordane	1.0	2.0	5.0	10	20	50	100	200
4,4'-DDE	1.0	2.0	5.0	10	20	50	100	200
Endosulfan I	1.0	2.0	5.0	10	20	50	100	200
Dieldrin	1.0	2.0	5.0	10	20	50	100	200
Endrin	1.0	2.0	5.0	10	20	50	100	200
4,4’-DDD	1.0	2.0	5.0	10	20	50	100	200
Endosulfan II	1.0	2.0	5.0	10	20	50	100	200
4,4’-DDT	1.0	2.0	5.0	10	20	50	100	200
Endrin Aldehyde	1.0	2.0	5.0	10	20	50	100	200
Methoxychlor	1.0	2.0	5.0	10	20	50	100	200
Endrin Sulfate	1.0	2.0	5.0	10	20	50	100	200
Endrin Ketone	1.0	2.0	5.0	10	20	50	100	200
Tetrachloro-m-xylene (surr)	1.0	2.0	5.0	10	20	50	100	200
Decachlorobiphenyl (surr)	1.0	2.0	5.0	10	20	50	100	200
Toxaphene	50	100	150	300	400	500	1000	
T-Chlordane	12.5	25	50	75	100	200	300	
Isodrin	0.5	1.0	2.0	5.0	10	20	40	
Mirex	0.5	1.0	2.0	5.0	10	20	40	
Diallate (total)	25	50	100	250	500	1000	2000	
Chlorobenzilate	25	50	100	250	500	1000	2000	
2,4’-DDT	2.5	5.0	10	50	100	200	400	
2.4’-DDD	2.5	5.0	10	50	100	200	400	
2,4’-DDE	2.5	5.0	10	50	100	200	400	

Table 5	
Column Degradation Evaluation Mix	
Component	Concentration ng/mL
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table 6							
LCS Matrix Spike and Surrogate Spike Levels							
	Aqueous (µg/L)			Soil (µg/kg)		Waste (µg/kg)	Air (µg/sample)
Analyte	Large Volume 1000mL → 10 mL	Reduced Volume 5mL		Large Volume 30g →10 mL	MAE 15g → 5mL	1g → 10mL	1 sample →10mL
		250mL →5 mL	125mL → 5 mL				
alpha-BHC	0.50	2.0	4.0	16.7	16.7	250	0.5
gamma-BHC (Lindane)	0.50	2.0	4.0	16.7	16.7	250	0.5
beta-BHC	0.50	2.0	4.0	16.7	16.7	250	0.5
delta-BHC	0.50	2.0	4.0	16.7	16.7	250	0.5
Heptachlor	0.50	2.0	4.0	16.7	16.7	250	0.5
Aldrin	0.50	2.0	4.0	16.7	16.7	250	0.5
Heptachlor Epoxide	0.50	2.0	4.0	16.7	16.7	250	0.5
gamma-Chlordane	0.50	2.0	4.0	16.7	16.7	250	0.5
alpha-Chlordane	0.50	2.0	4.0	16.7	16.7	250	0.5
4,4-DDE	0.50	2.0	4.0	16.7	16.7	250	0.5
Endosulfan I	0.50	2.0	4.0	16.7	16.7	250	0.5
Dieldrin	0.50	2.0	4.0	16.7	16.7	250	0.5
Endrin	0.50	2.0	4.0	16.7	16.7	250	0.5
4,4'-DDD	0.50	2.0	4.0	16.7	16.7	250	0.5
Endosulfan II	0.50	2.0	4.0	16.7	16.7	250	0.5
4,4'-DDT	0.50	2.0	4.0	16.7	16.7	250	0.5
Endrin Aldehyde	0.50	2.0	4.0	16.7	16.7	250	0.5
Methoxychlor	0.50	2.0	4.0	16.7	16.7	250	0.5
Endrin Sulfate	0.50	2.0	4.0	16.7	16.7	250	0.5
Endrin Ketone	0.50	2.0	4.0	16.7	16.7	250	0.5
Tetrachloro-m-xylene (surr)	0.20	0.80	1.6	6.67	6.67	100	0.2
Decachlorobiphenyl (surr)	0.20	0.80	1.6	6.67	6.67	100	0.2
Toxaphene	5.0	20.0	40.0	167	167	2500	5

Table 7—QC Acceptance Criteria—Method 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for X (µg/L)	Range for P, P_s(%)
Aldrin	2.0	0.42	1.08-2.24	42-122
α-BHC	2.0	0.48	0.98-2.44	37-134
β-BHC	2.0	0.64	0.78-2.60	17-147
δ-BHC	2.0	0.72	1.01-2.37	19-140
γ-BHC	2.0	0.46	0.86-2.32	32-127
Chlordane	50	10.0	27.6-54.3	45-119
4,4″-DDD	10	2.8	4.8-12.6	31-141
4,4″-DDE	2.0	0.55	1.08-2.60	30-145
4,4′-DDT	10	3.6	4.6-13.7	25-160
Dieldrin	2.0	0.76	1.15-2.49	36-146
Endosulfan I	2.0	0.49	1.14-2.82	45-153
Endosulfan II	10	6.1	2.2-17.1	D-202
Endosulfan Sulfate	10	2.7	3.8-13.2	26-144
Endrin	10	3.7	5.1-12.6	30-147
Heptachlor	2.0	0.40	0.86-2.00	34-111
Heptachlor epoxide	2.0	0.41	1.13-2.63	37-142
Toxaphene	50.0	12.7	27.8-55.6	41-126

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).

X = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).

P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2).

D = Detected; result must be greater than zero.

Note: These criteria are based directly upon the method performance data in Table 8. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 8.

Table 8—Method Accuracy and Precision as Functions of Concentration—Method 608

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
Aldrin	0.81C+0.04	0.16X−0.04	0.20X−0.01
α-BHC	0.84C+0.03	0.13X+0.04	0.23X−0.00
β-BHC	0.81C+0.07	0.22X−0.02	0.33X−0.05
δ-BHC	0.81C+0.07	0.18X+0.09	0.25X+0.03
γ-BHC	0.82C−0.05	0.12X+0.06	0.22X+0.04
Chlordane	0.82C−0.04	0.13X+0.13	0.18X+0.18
4,4′-DDD	0.84C+0.30	0.20X−0.18	0.27X−0.14
4,4′-DDE	0.85C+0.14	0.13X+0.06	0.28X−0.09
4,4′-DDT	0.93C−0.13	0.17X+0.39	0.31X−0.21
Dieldrin	0.90C+0.02	0.12X+0.19	0.16X+0.16
Endosulfan I	0.97C+0.04	0.10X+0.07	0.18X+0.08
Endosulfan II	0.93C+0.34	0.41X—0.65	0.47X−0.20
Endosulfan Sulfate	0.89C−0.37	0.13X+0.33	0.24X+0.35
Endrin	0.89C−0.04	0.20X+0.25	0.24X+0.25
Heptachlor	0.69C+0.04	0.06X+0.13	0.16X+0.08
Heptachlor epoxide	0.89C+0.10	0.18X−0.11	0.25X−0.08
Toxaphene	0.80C+1.74	0.09X+3.20	0.20X+0.22

X′ = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration found of X, in µg/L.

S′ = Expected interlaboratory standard deviation of measurements at an average concentration found of X, in µg/L.

C = True value for the concentration, in µg/L.

Table 9 Summary of Method 608 QC Requirements

QC Objective	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Prior to sample analysis; as needed thereafter when CCV fails or major maintenance; column change	<10% RSD to use average RF; Regression curve if >10% RSD	Re-prep and re-curve
Calibration Curve Verification (CCV)	Each work day prior to sample analysis	± 15% of the expected response	Repeat once using a fresh calibration standard; New calibration curve.
Matrix Spike (MS)	10% of the samples from each sample site	Compare recovery (P) with acceptance criteria in Table 7	Analyze QC Check Standard (QCCS) and compare recovery (Ps) with acceptance criteria in Table 7.
Quality Control Check Standard (QCCS)	Analyzed if any analyte in matrix spike is outside QC acceptance limit.	Compare recovery (Ps) with acceptance criteria in Table 7	Recovery for the QCCS is evaluated for any parameter that is outside the QC acceptance criteria in the matrix spike (MS). I fa parameter is outside acceptance criteria in both the MS and QCCS, that parameter can not be reported for regulatory purposes.

Table 10
Suggested Analytical Sequence

Initial Calibration, including ICV

Solvent blank (optional)
Breakdown Mix (PEM)
ICAL

Individual Mix AB	All levels and ICV
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]

Up to 20 samples (unless 12 hours comes first)
Closing/Bracketing CCVs

Individual Mix AB	Level 5 - Mid level (Continuing calibration)
Technical Chlordane	Level 4
Toxaphene	Level 4

If Less than12 hours has elapsed since initial PEM:

Then Continue with up to 20 samples or 12 hours
Closing/Bracketing CCVs

Individual Mix AB	Mid level (Continuing calibration)
Technical Chlordane	Level 4
Toxaphene	Level 4

Else Breakdown Mix (PEM)
Opening CCVs

Individual Mix AB	Level 4 - Alternative Mid level (Continuing calibration)
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]

Up to 20 samples (unless 12 hours comes first)
Closing CCVs

Individual Mix AB	Level 5 - Mid level (Continuing calibration)
Technical Chlordane	Level 4
Toxaphene	Level 4

[1] A multi-point curve for any of the multi-component analytes may be included

Continuing Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

Table 11
Recommended Maintenance for Method 8081 Instruments:

1. For failing CCV's:
   - Turn off inlet temperature and oven temperature. Wait until you feel comfortable handling the pieces. Note: The inside nut where the gold seal sits is always warmer then anything else in the oven.
   - Before opening the system, turn off the carrier gas. This should introduce minimal air into the system, and means the detectors can remain at their operating temperature.
   - Do a "full front inlet" maintenance routine, which includes changing liner, septa, gold seal and trimming the guard column by a half-loop to a loop.
   - Always work quickly, but there is no need to rush. While less time open is better, completing the maintenance within 10-15 minutes is acceptable.
   - The ideal column length out of the top of the ferrule is 5mm.
   - Once maintenance is done, check for leaks, then bake the oven at 315 for a few cycle times (this means 30 minutes to an hour).
   - NEVER bake the inlet, as this can warp the septa and cause a leak, negating any positive results of maintenance.
   - When the bake cycles are completed, prime the system with expired high-level calibration standards (level 6 or higher), then run a blank, PEM and opening calibration verification to see if previous ICAL holds.
2. For failing PEM
   - If the CCV's are passing, but the breakdown is going high (typically Endrin) then a simple liner and septa change may be sufficient.
   - If the liner and septa change isn't enough, then replace the gold seal and trim the guard column as well ("full front inlet", as discussed above).
   - Also verify the last time that carrier gas filters and split vent traps were changed. These may also have an effect on breakdown.
3. Time to ICAL
   - If PEM's are passing great, but CCV's just don't want to pass on an ICAL that has been around for over a week, it's probably time to ICAL. (No maintenance needed).
   - If maintenance has been done, and the opening CCVs still don't pass, it's time to perform initial calibration.
   - Sometimes, a few (about 5) sample injections are necessary to prime the system prior to calibration. Use previously analyzed samples, then load the high-level standards, a couple of blanks, and the initial calibration sequence.

Title: Chromatographic Analysis Based on SW-846 Methods 8000B/8000C, 8082/8082A, Method 608, and Compendium Methods TO-4A, and TO-10A [Methods 8000B, 8000C, 8082, 8082A, 608, TO-4A, TO-10A]

Approvals (Signature/Date):

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures to be used when SW-846 Method 8000B or Method 8000C is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This SOP is to be applied when SW-8/46 Methods 8082 or 8082A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOP, WS-OP-0002. The PCBs are determined and quantitated as Aroclor mixtures.

This procedure is also applicable to the determination of PCBs in municipal and industrial discharges under 40 CFR 136.1.

1.2. Table 1 lists Aroclor mixtures which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. Matrix interferences may result in higher RLs than those listed.

Note: This SOP does not include directions for congener specific analysis

1.3. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. In general, aqueous samples are prepared for analysis using separatory funnel liquid-liquid extraction, and semivolatile analytes in solid samples are prepared using sonication or microwave assisted extraction.

2.2. This SOP presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP WS-OP-0002.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, as outlined in the preparation SOP WS-OP-0002.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The PCB solutions used to prepare standards for this method may arrive at concentrations as high as 1,000 ppm. Exercise extreme caution when handling these high concentration standards.

5.1.2. If any PCB standards are spilled inside or outside of a fume hood at concentrations equal to or greater than 5 PPM, all materials in contact must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS Coordinator for specific disposal instructions <u>before</u> cleaning up the spill.

5.1.3. If any PCB standards are spilled outside the fume hood at concentrations greater than 200 ppm, the Emergency Response Team must be activated for special clean-up procedures, and possibly additional testing of the spill location and additional regulatory notifications.

5.1.4. All glassware and lab trash in contact with any PCB's at concentrations equal to or greater than 5 PPM must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS Coordinator for specific disposal

instructions before disposing of this waste.

5.1.5. The autovials used on the instrument contain a small amount of solvent after the analysis is completed, which can evaporate into the air through the puncture in the septa. These autovials may be collected in an open tripour or similar cup at the instrument while the analyst is removing them from the tray, but once that is complete the vials must be transferred to a waste container in a fume hood. They cannot be left out on the bench by the instrument.

5.1.6. Ensure that the outlet lines from the instrument are secured and lead into either a fume hood or inlet point to the fume hood exhaust system.

5.1.7. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.1.8. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing the EH&S coordinator shall be immediately notified and a letter sent to the NRC or local state agency.

5.1.9. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when working with open sample extracts, spikes, surrogates or solvents. Latex or vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should only be used when working with closed extract vials.

5.1.10. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.11. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the

method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2. Gas Chromatograph - An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. A data system capable of measuring peak area and/or height is required. The GC injection port must be designed for capillary columns.

6.3. This laboratory utilizes an Agilent 6890 gas chromatograph with ChemStation D.02.00.275 operating system or equivalent and Chrom Peak Review version 2.1 data acquisition system or equivalent.

6.4. A ^{63}Ni electron capture detector is required.

6.5. Refer to Table 2 for analytical columns.

6.6. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Standards

7.1.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampule is opened, if the standard is supplied in a sealed ampule. If a vendor supplied standard has an earlier

expiration date then that date is used.

7.1.2. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standard solutions are stored at ≤ 6ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using. Stock standard solutions must be replaced after one year.

7.1.3. Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at ≤ 6ºC and protected from light. The standards must be replaced at least every six months or sooner if comparisons with check standards indicates a problem.

7.1.4. Refer to Table 3 for details of calibration standards.

7.2. Gases for carrier and make-up: Hydrogen, Helium, and Nitrogen.

7.3. Solvent for dilutions (if needed) and syringe cleaning: Hexane, pesticide grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Extracts must be refrigerated at ≤ 6ºC and analyzed within 40 days of the end of the extraction.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. The minimum initial demonstration required is analysis of a single point calibration.

9.2. Quality Control Batch

9.2.1. The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In

some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch may contain multiple MS/MSDs. Method 608 requires an LCS frequency equivalent to 10% of all samples analyzed.

9.2.2. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible. QC samples should be analyzed with the field samples, however, field samples may be split between instruments or sequences as needed.

9.2.3. If a QC batch is analyzed on more than one instrument, at the minimum an instrument blank must be analyzed on the second instrument to demonstrate that the system is free of interferences.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). Refer to policy WS-PQA-003 for more details.

9.3.1. These limits do not apply to dilutions. Surrogate and matrix spike recoveries will be reported in dilutions greater than 5x will be qualified.

9.3.2. All surrogate, LCS, and MS recoveries must be entered into TALS or other database so that accurate historical control limits can be generated.

9.4. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if batch precision data is required by the client or program. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented.

9.5. Surrogates

Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Reprepare and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem. Repreparation is not necessary if there is obvious chromatographic interference or other evidence of matrix impacts.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate

that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect

9.5.1. While samples may be spiked with both TCX and DCB as surrogates, only DCB is used for control purposes. If DCB does not meet the recovery criteria, corrective action must occur.

9.5.2. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12) unless one column is out of control, in which case the in-control result is reported. For DOD projects, report the surrogate value from the primary column, rather than the higher value.

9.5.3. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.5.4. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.6. Method Blanks

One method blank must be processed with each preparation batch. The method blank consists of reagent water for aqueous samples, and sodium sulfate for solid samples (refer to the appropriate preparation SOP for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

Certain programs may require a more stringent evaluation of the method blank. For instance, DOD requires that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.

- Re-preparation and reanalysis of any samples with reportable concentrations of analytes less that 10 times the value found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. This must be documented in the NCM program.
- If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.6.1. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.7. Laboratory Control Samples (LCS)

9.7.1. For each batch of samples, analyze a LCS. At a minimum, the LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes if so required by the client or program governing the samples, such as DOD. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur.

9.7.2. If dual column analysis is used the choice of which result to report is described in Section 12. For DOD projects, report the value from the primary column, rather than the higher value.

9.7.3. Generally, Aroclors 1016 and 1260 are spiked for the LCS and MS/MSD. Other Aroclors may be spiked as requires by regulatory programs or client requirements. Spiking levels are described in Table 4.

9.7.4. Each Aroclor must be spiked and its recovery measured in at least 2 LCS over a 2 year time period.

9.7.5. LCS recovery for Method 8082/8082A limits and RPD's are set by in-house studies (see WS-PQA-003). The recovery limits for methods TO-4A and TO10A are given in Table 6.

9.7.6. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:

- Check calculations
- Check instrument performance
- Reanalyze the LCS, and if still outside of control limits
- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.7.7. The analyst should evaluate the anomalous analyte recovery for possible trends, as described in the QC Program document (WS-PQA-003). Control charts will be used to track trends and sporadic marginal exceedances.

9.7.8. Data may be reported with an anomaly in the following cases:

- The LCS recoveries are high and the analyte of concern is not detected in field samples.

- All target requested analytes are within control, but other LCS compounds are out of control.

9.7.9. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

9.7.10. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.7.11. If the batch is re-extracted past sample holding time, both sets of results should be reported, flagged and narrated.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Method 608 requires a matrix spike on at least 10% of the samples from each sample site being monitored. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits, as described in the QC Program document (WS-PQA-003). See Table 6 for TO-4A and TO-10A limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed.
- The MS/MSD must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report is described in Section 12. For DOD projects, report the value from the primary column, rather than the higher value.

9.8.2. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. File an NCM stating that the 4X rule was applied. This NCM must be included in the final report.

9.8.3. For Method 608, the spike concentration for each parameter is found in Table 7. Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria. If the spike concentration was lower than the concentration in Table 7, the acceptance criteria must still be met or use the optional acceptance criteria in Table 8. .

9.8.3.1. If the recovery (P) is outside the acceptance criteria in Table 7 a QC check standard (QCCS / LCS) is analyzed and the percent recovery (Ps) is compared to Table 7. Analytes that fail in the matrix spikes and QCCS / LCS) are out of control and can not be reported for regulatory compliance purposes.

9.9. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.11. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

9.12. While samples may be spiked with both TCX and DCB as surrogates, only DCB is used for control purposes. If DCB does not meet the recovery criteria, corrective action must occur.

10. CALIBRATION

10.1. Initial Calibration

10.1.1. Refer to Table 5 for the initial calibration analytical sequence.

10.1.2. A multi point calibration (6 or 7 points are typical) of the Aroclor 1016/1260 mix is generated, and only mid level single points for the other Aroclor mixes are analyzed.

10.1.2.1. For Methods 8082, 8082A, TO-4A and TO-10A, the average response factor is used to quantitate Aroclors 1260 and 1016; other Aroclors are quantitated from the single point.

10.1.2.2. In the case of Method 608, quantitation of Aroclors must be performed using initial calibrations consisting of at least 3 levels.

10.1.2.3. In the case of work which falls under DoD QSM 4.x, quantitation of Aroclors must be performed using initial calibrations consisting of at least 5 levels.

10.1.2.4. The analyst may include a full calibration (including ICV) for any of the Aroclors with the initial calibration, if requested by the client or program.

10.1.2.5. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the multi-point calibration rather than the 1016/1260 mix.

10.1.3. The high and low standards for the initial multi-point calibration of 1016/1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration the extract must be diluted and reanalyzed.

10.1.4. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.1.5. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new (uncalibrated) columns, or replacing the ECD detector. A new calibration is not required after performing column maintenance, replacing the septum or syringe, or performing other minor maintenance.

10.1.6. With the exception of instances detailed in policy CA-T-P-002, Selection of Calibration Points, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Curve fitting other than average response factor requires a multipoint calibration for each Aroclor mixture.

10.1.7. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak response to the mass or concentration injected may be used to prepare a calibration curve. Peak response is generally measured as the area of the peak. The choice of height or area is made at the time of initial calibration, and may not be altered for a given

compound until the next initial calibration.

Equation 1

$$\text{Response Factor (RF)} = \frac{\text{Peak Area}}{\text{Concentration of Solution (ng/ml)}}$$

10.1.8. To calibrate Aroclor mixtures, select 3-5 major peaks in the analyte pattern. At least 3 peaks are required. Each selected peak should be at least 25% of the largest Aroclor peak in the pattern. The set of peaks chosen should include at least one peak unique to that Aroclor. Calculate a response factor (Equation 1) for each of the chosen peaks.

10.1.9. The center of the retention time window for each peak is updated with the initial calibration. If more than one calibration level is used for an Aroclor (such as with the 1016/1260), use the mid-level (level 4 from Table 3) standard.

10.2. Calibration Curve Fits

Curve fits other than average response factor require a multi-point calibration for each Aroclor mixture. Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the % RSD of the response factor or calibration factor of each analyte is ≤ 20%. Method 608 requires the %RSD to be <10% for the average response factor. If an analyte exceeds the criteria, then a regression line (linear or curved) should be attempted. If the regression lines do not meet criteria (coefficient of determination, r2 ≥ 0.990, or correlation coefficient, r ≥ 0.995), evaluate the calibration, then perform any required instrument maintenance prior to repeating the initial calibration process.

Note: If a regression line is used, multi-level calibrations must be performed for each Aroclor, as the assumption of linearity through the origin for each Aroclor is no longer valid.

10.2.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.2.2. Average response factor - The average response factor may be used for an analyte if the percent relative standard deviation (%RSD) of the response factors for a given analyte is ≤ 20% (< 10% for Method 608). The equation for average response factor is:

Equation 2

$$\overleftarrow{\text{RF}} = \frac{\sum_{i=1}^{n} RF_i}{\text{n}}$$

Where n = Number of calibration levels, and

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.2.3. Linear regression

The linear fit uses the following function

Equation 2 $y = ax + b$ **or** $x = \frac{(y-b)}{a}$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.2.4. Quadratic curve

The quadratic curve uses the following function:

Equation 3 $y = ax^2 + bx + c$

Where: y = Instrument response
x = Concentration
a = Curvature
b = Slope
c = Intercept

10.3. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve fit (linear or otherwise) is used, the intercept of the curve at zero response must be less than ± ½ the reporting limit for the analyte.
- For the linear case, the correlation coefficient (r) must be greater than or equal to 0.995. For the quadratic case the coefficient of determination (r^2) may be used, and must be greater or equal to 0.990.

10.4. Initial Calibration Verification (ICV): A second source standard must be analyzed with the initial calibration curve. Each compound of the second source calibration must be within ± 20% (Method 8082A) or within ± 15% (Methods 608, 8082, TO-4A, and TO-10A) of its expected value when calculated against the initial calibration. Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards
- Recalibrate as needed

10.5. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If calibrations are required, calibration data quality objectives may need to be established with the client.

10.6. Calibration Verification

10.6.1. At a minimum, the working calibration curve or RF must be verified by the analysis of a mid-level calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The working calibration curve or calibration factor for Method 608 must be verified on each working day prior to sample analysis.

10.6.1.1. Methods 8082 and 8082A requires verification at the beginning, after every 12 hours or 20 samples (whichever is more frequent), and at the end of the analysis sequence.

10.6.1.2. Some programs, such as DOD, may require more frequent verification, or base the frequency on the number of samples instead of a time period. DOD requires a CCV for every ten injections of field samples.

10.6.1.3. The initial calibration verification standard used at the start of the analysis sequence must be a different concentration than the mid-level calibration standard used for subsequent calibration verifications. Recommended concentrations are given in Table 3.

10.6.1.4. At a minimum, the calibration verification includes analysis of the Aroclor 1260/1016 mix.

10.6.1.5. It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific Aroclor is expected, it should be included in the daily calibration check.

10.6.2. % Difference is used to compare the continuing calibration verification to the initial calibration when the average response factor is used.

Equation 5

$$\%Difference = \frac{\left|X - \overline{X}\right|}{X} \times 100\%$$

Where X = Calibration or response factor of the CCV

$\overline{X}$ = Average Calibration or response factor.

10.6.3. % Drift is used for comparing the continuing calibration to a liner or quadratic curve. The criterion for % drift is the same as for % difference.

Equation 4

$$\%\text{Drift} = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

10.6.4. Any individual compounds with %D ≤ 20% (Method 8082A) or within ≤ 15% (Methods 8082, 608, TO-4A, and TO-10A) meet the calibration criteria. Some programs such as DOD may have other %D requirements.

10.6.5. It is not necessary to run a daily continuing calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration, i.e., the calibration curve and second source standard (initial calibration verification).

10.6.6. Samples must be bracketed by calibration verification standards that meet the criteria listed above.

10.6.7. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria and the preceding samples show no evidence of similar failure, the preceding samples have been successfully bracketed and no further action is required. If this CCV fails or adjustments to the instrument are performed before the repeat CCV then the preceding samples have not been successfully bracketed but analysis may continue.

10.6.8. It is not allowed to analyze repeat CCVs on unattended runs.

10.6.9. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.6.10. Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 20% (Method 8082A) or ± 15% (Methods 8082, TO-4A, and TO-10A) (or other specified requirements) corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than the criteria above, (or other specified requirements) a new calibration curve may be necessary.

10.6.10.1. Any samples injected after the last good continuing calibration standard must be reinjected, unless one of the following conditions is satisfied:

10.6.10.2. If the CCV response is elevated and the preceding samples are non-detect for all analytes of concern, an NCM may be filed and the data reported.

10.6.10.3. If the samples have already been analyzed twice, and it is apparent that matrix effects are causing the out of control event, an NCM may be filed and the data reported. Client consultation or dilution of samples may be necessary.

11. PROCEDURE

11.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified.
The Nonconformance memo will be filed in the project file. Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Extraction and cleanup – Extraction and cleanup procedures are described in SOP WS-OP-0002.

11.3. Suggested gas chromatographic conditions are given in Table 2.

11.4. Allow extracts to warm to ambient temperature before injection. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence
An analytical sequence starts with an initial calibration or a daily calibration. The suggested analytical sequence is given in Table 5.

11.5.1. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a CCV.

11.5.2. Solvent blanks may be analyzed immediately following CCVs or suspect samples at any point during the run, however, they may not be routinely used to "clean out the instrument" immediately prior to CCVs.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte. For Aroclors use the retention time of the peaks used for calibration. The retention time window is defined as plus or minus three times the standard deviation of the retention times of each analyte.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid point of the initial calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than ± 0.03 minutes, use ± 0.03 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by initial calibration.

11.7. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client.

Percent moisture must be determined if results will be reported as dry weight. Refer to SOP WS-OP-0013 for determination of percent moisture.

12. CALCULATIONIS/DATA REDUCTION

12.1. Identification of Aroclors

12.1.1. Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.1.2. Second column confirmation of Aroclors will only be performed when requested by the client, or if the pattern is not clear or there is no historical data leading to a suspicion that Aroclors may be present. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence. Certain programs, such as the DOD QSM, require second column confirmation of Aroclors.

12.1.3. If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect, and the results are qualified. If no anomalies, such as overlapping peaks, are noted, report the higher result, or both results.

12.2. Quantitation of Aroclors

12.2.1. If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1216 and 1232 is present, then only the predominant Aroclor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative.

12.2.2. If well-separated Aroclor patterns are present, then both Aroclors are quantitated and reported.

12.2.3. Certain programs, such as those under the DOD QSM, may require that multiple mixtures be reported if identified. Refer to the DOD QSM policy WS-PQA-021 for further details.

12.3. While both decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) may be spiked into the extracts, the monitored surrogate for this method is DCB. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB). Corrective action is necessary if DCB is outside of acceptance limits. TCX may be requested and controlled on a project specific basis.

12.4. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix or screening results.

12.5. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.5.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.5.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request. Refer to policy WS-PQA-021 for requirements for projects under the auspices of the DOD.

12.6. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in a nonconformance memo.

12.7. Dual channel quantitation – In the event that two columns are used for the analysis, used the guidance below to determine how to report the data. Certain programs, such as DOD, may have differing requirement for the reporting data from dual channel analyses. Refer to the DOD QSM policy (WS-PQA-021) for DOD requirements, or the client QAPPs.

For confirmed results, two approaches are available to the analyst:

A) The primary column approach
B) The better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.7.1. Primary column approach (A):

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true:

- There is obvious chromatographic interference on the primary column.
- The result on the secondary column is 40% greater than the result on the primary column, and there is no reason apparent on the chromatogram for rejecting the higher value (i.e., faulty integration, negative dips affecting the baseline, etc.)
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for reanalysis.) In this instance, only ND (not requiring deconfirmation) or spike results may be reported.
- LCS, surrogates, and MS/MSD are reported from the primary column unless a standard failure dictates reporting a given analyte from the secondary column. This must be noted in an anomaly.

12.7.2. Better result approach (B):

The higher of the two results is normally reported unless obvious chromatographic interference is noted.

LCS, surrogates, and MS/MSD are reported from the primary column unless a standard failure dictates reporting a given analyte from the secondary column. This must be noted in an anomaly.

12.7.3. If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect, and the results are qualified. If no anomalies, such as overlapping peaks, are noted, report the higher result, or both results. RPD is calculated using the following formula:

Equation 7

$$RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\%$$

Where R = Result

12.7.4. The experience of the analyst should weigh the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.8. Calculations

12.8.1. Concentration in the extract (single peaks):

Equation 8

$$Concentration(ng/mL) = \frac{A}{CF}$$

Where: A = Response for the analyte in the sample
CF = Calibration factor. Area (ng/mL), Section 10.1

12.8.2. Concentration in the extract, Aroclor mixture:

Equation 9

$$Concentration(ng/mL) = \frac{\sum_{i=1}^{n} C_{peak}}{n}$$

Where: C_{peak} = Concentration of the peak, using Equation 8
n = number of peaks for the Aroclor mixture

12.8.3. Concentration in the sample

Equation 10

$$Concentration(ug/L\ or\ ug/kg) = \frac{C_{ex}\ x\ D\ x\ FV}{A_{sa}}$$

Where: C_{ex} = Concentration in the extract (Equations 8 and 9)
D = Dilution factor

FV = Final extract volume (mL)
A_{sa} = Amount of sample extracted (mL or g)

12.8.4. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:.

Equation 11

$$\%\ \mathrm{Recovery} = \frac{Concentration\ \ found}{Concentration\ spiked}\ x\,100$$

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes

for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. 1-2 milliliter autovials containing small amounts of solvent extract. These are collected at the instrument and immediately transferred to a satellite collection container in a fume hood. When full or after no more than one year, these satellite collection containers are transferred to the waste disposal area for disposal.

15.2. Extracts, extract dilutions, all disposable materials (filters, disposable glassware, etc.) with concentrations of Aroclors greater than 50 ppm. These must receive special handling, shipping and disposal as PCB waste. Contact the Hazardous Waste Technician for specific instructions, as these cannot be disposed in normal waste streams.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, Method 8000B, Revision 0, December 1996, Determinative Chromatographic Separations

16.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, Method 8000C Revision 3, March 2003, Determinative Chromatographic Separations

16.3. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, Method 8082, Revision 0, December 1996, Polychlorinated Biphenyls (PCBs) by Gas Chromatography

16.4. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-4A, January 1999, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)

16.5. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-10A, January 1999, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)

16.6. 40 CFR Appendix A Part 136 Method 608 April 8, 2014.

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Modifications from Reference Method 8082

17.2.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.3. Modifications from Reference Methods TO-4A and TO-10A

17.3.1. A minimum 5-point calibration is performed for Aroclor 1016/1260, and a single point calibration is performed for the remaining mixtures. An average response factor is determined for each analyte.

17.3.2. The calibration is verified daily or every 12 hours with the mid point standard.

17.3.3. The RT windows are set using the mid point standard and are updated daily or every 12 hours from the mid point standard.

17.3.4. Sample analysis is performed using a DB-608 and DB-1701, or other appropriate GC columns.

17.3.5. Instrument operating conditions will be determined on an instrument and column specific basis.

17.3.6. Method Blanks are deemed acceptable if no target analytes are present at or above the reporting limits or >10% of the measured concentration in the associated samples.

17.3.7. Sample results are not corrected for extraction efficiency, as determined by surrogate recovery.

18. ATTACHMENTS

18.1. Table 1 – Standard Analyte List and Reporting Limits

18.2. Table 2 – Recommended Conditions for DB-608, DB5 OR DB-1701

18.3. Table 3 – Calibration Levels

18.4. Table 4 – LCS/Matrix Spike and Surrogate Spike Levels for Aroclor Analysis with Acid Cleanup

18.5. Table 5 – Suggested Analytical Sequence

18.6. Table 6– QC Acceptance Criteria – Methods TO-4A and TO-10A

18.7. Table 7 - QC Acceptance Criteria – Method 608

18.8. Table 8– Method Accuracy and Precision as Functions of Concentrations – Method 608

18.9. Table 9– Summary of Method 608 QC Requirements.

19. REVISION HISTORY

19.1. WS-GC-0002, Revision 5.0, Effective 04/15/2014

19.1.1. Section 1.1, appended, "This procedure is also applicable to the determination of PCBs in municipal and industrial discharges under 40 CFR 136.1."

19.1.2. Section 9.2.1, inserted, "Method 608 requires an LCS frequency equivalent to 10% of all samples analyzed."

19.1.3. Section 9.8, inserted "Method 608 requires a matrix spike on at least 10% of the samples from each sample site being monitored."

19.1.4. Added Section 9.8.3 and 9.8.3.1 regarding the spiking criteria for Method 608

19.1.5. Inserted Section 10.1.2.1, 10.1.2.2, and 10.1.2.3, which discuss the calibration curve set-up and criteria for the various references methods associated with this SOP.

19.1.6. Section 10.2, inserted, "Method 608 requires the %RSD to be <10% for the average response factor."

19.1.7. Section 10.2, appended, "Note: If a regression line is used, multi-level calibrations must be performed for each Aroclor, as the assumption of linearity through the origin for each Aroclor is no longer valid."

19.1.8. Section 10.2.2, first sentence, appended, "(< 10% for Method 608)."

19.1.9. Section 10.6.1, appended, " The working calibration curve or calibration factor for Method 608 must be verified on each working day prior to sample analysis."

19.1.10. Added Section 16.1, "40 CFR Appendix A Part 136 Method 608 April 8, 2014."

19.1.11. Added Tables 7, 8, and 9

19.2. WS-GC-0002 rev 4.6, Effective 07/03/2013

19.2.1. Amended Section 5 to accommodate the changes in source standards and resulting TSCA requirements.

19.2.2. Updated SOP to reflect change to TALS system

19.2.3. Updated to include Method 8000C/8082A criteria. Specific sections changed are the title and Sections 1.1, 9.7.5, 10.4, 10.6.4, and 10.6.10

19.3. WS-GC-0002 rev. 4.5 Effective 04/12/2013

19.3.1. Changed references from 'Target' to 'Chrom' and from 'Quantims' to 'TALS'.

19.3.2. Updated Tables 1, 2, and 3 with current operating parameters.

19.3.3. Editorial revisions.

19.4. WS-GC-0002 r4.4, Effective 02/17/2012

19.4.1. Section 11.6.3: Changed retention time window from 0.05 minutes to 0.03 minutes.

19.4.2. Editorial revisions.

19.5. WS-GC-0002 r4.3, Effective -1/07/2011

19.5.1. All references to QSM 4.1 were updated to QSM 4.x to encompass new revisions.

19.5.2. Changed Section 10.1.8 from "…select 5-10 major peaks in the analyte pattern. At least 5 peaks are required." to "…select 3-5 major peaks in the analyte pattern. At least 3 peaks are required."

Attachments

Table 1 Standard Analyte List and Reporting Limits					
Compound	Reporting Limit				
	Water µg/L	Soil µg/kg	Waste µg/kg	Air µg/sample	Wipe µg/sample
Aroclor 1016	1.0	33	1000	0.75	1.0
Aroclor 1221	1.0	33	1000	0.75	1.0
Aroclor 1232	1.0	33	1000	0.75	1.0
Aroclor 1242	1.0	33	1000	0.75	1.0
Aroclor 1248	1.0	33	1000	0.75	1.0
Aroclor 1254	1.0	33	1000	0.75	1.0
Aroclor 1260	1.0	33	1000	0.75	1.0
Aroclor 1262	1.0	33	1000	0.75	1.0
Aroclor 1268	1.0	33	1000	0.75	1.0

The following concentration factors are assumed in calculating the Reporting Limits:

Table 1B			
Matrix	Sample Volume	Final Extract Volume	Initial Extract Cleaned and submitted to GC group
Water	1000 mL	10 mL	2 mL
Water by Reduced Volume Extraction (RVE)	250mL	5 mL	2 mL
Low Level Soil/Solid	30 g	10 mL	2 mL
Low Level Soil/Solid by EPA 3586 (MAE)	15 g	5 mL	2 mL
High Level Solid/Waste Oil	1 g	10 mL	2 mL
Air (trapped in PUFF or similar)	1 Sample	10 mL:	2 mL

Table 2	
Parameter	**Recommended Conditions for DB-608, DB5 or DB-1701**
Injection port temp	230°C
Detector temp	300°C
Temperature program	180°C for 1 minute, 8°C /minutes to 280°C, hold 10 minutes
Column 1	DB-608 30m x 0.53mm x 0.83µm
Column 2	DB-1701 30m x 0.53mm x 1.0µm
Column 3	DB-5 30m x 0.53mm x 1.0µm
Injection	2µL
Carrier gas	Hydrogen
Make up gas	Nitrogen
Y splitter	Restek
	Recommended Conditions for Stx-CLPesticides 1 and 2
Injection port	230°C, 70.0 psi for 0.5 min in pulsed splitless mode
Detector temp	300°C
Temperature program	100°C for 1 minute - 12.5°C /minutes to 300°C, hold 2 minutes - 30°C /minutes to 320°C, hold 1.33 minutes
Flow program	2.8 mL/min for 19 min, 30 mL/min^2 to 15 mL/min for 1.6 min
Column 1	Stx-CLPesticides-1 30m x 0.32mm x 0.5µm
Column 2	Stx-CLPesticides-2 30m x 0.32mm x 0.25µm
Injection	2µL
Carrier gas	Hydrogen
Make up gas	Nitrogen
Y splitter	Restek

Table 3							
Calibration Levels ng/mL							
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**
Aroclor 1016/1260	25	50	100	200	500	1000	2000
Aroclor 1242	25	50	100	200	500	1000	2000
Aroclor 1221	25	50	100	200	500	1000	2000
Aroclor 1254	25	50	100	200	500	1000	2000
Aroclor 1232	25	50	100	200	500	1000	2000
Aroclor 1248	25	50	100	200	500	1000	2000
Aroclor 1262	25	50	100	200	500	1000	2000
Aroclor 1268	25	50	100	200	500	1000	2000
Surrogates are included with all the calibration mixes at the following levels:							
Tetrachloro-m-xylene	2.5	5	10	20	50	100	200
Decachlorobiphenyl	2.5	5	10	20	50	100	200

Note: It is recommended to use the Level 4 standard as the initial CCV of an analytical sequence (as described in Section 10 of this SOP), and the Level 5 for subsequent CCVs.

Table 4					
LCS/Matrix Spike and Surrogate Spike Levels for Aroclor Analysis with Acid Cleanup					
	Aqueous µg/L	Soil µg/kg	Waste µg/kg	Air µg/sample	Wipes µg/sample
Aroclor 1016	2.0	66.7	2000	2.0	2.0
Aroclor 1260	2.0	66.7	2000	2.0	2.0
Tetrachloro-m-xylene (Surrogate)	0.20	13.3	400	0.2	0.4
Decachlorobiphenyl (Surrogate)	0.20	13.3	400	0.2	0.4

Table 5
Suggested Analytical Sequence (Method 8082/TO-4A/TO-10A)

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1016/1260	Level 6
8	Aroclor 1016/1260	Level 7
9	Aroclor 1016/1260	ICV Level 6 (1000 ppb)
10	Solvent Blank	
11	Aroclor 1221/1254	Level 4
12	Aroclor 1232/1262	Level 4
13	Aroclor 1242/1268	Level 4
14	Aroclor 1248	Level 4
15	Aroclor 1221/1254	Level 4
16	Solvent blank	
17-33	Sample 1-20 (or as many samples as can be analyzed in 12 hours)	
34	Aroclor 1016/1260	Level 4
etc		

Daily Analysis

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 3
3-22	Sample 1-20 (or as many samples as can be analyzed in 12 hours)	
23	Aroclor 1016/1260	Level 4
24-43	Samples 21-40 (or as many samples as can be analyzed in 12 hours)	
44	Aroclor 1016/1260	Level 4
etc		

Note: Some programs and clients require that CCVs be analyzed after every 10 samples, rather than every 12 hours.

TABLE 6
LCS and Surrogate Recovery Limits

	LIMITS		
Method	LCS Native Recoveries	Surrogate	RPD
TO-4A	65 – 125	60 – 120	30%
TO-10A	65 – 125	60 – 120	30%

Table 7
QC Acceptance Criteria—Method 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for X (µg/L)	Range for P, P_s(%)
PCB-1016	50	10.0	30.5-51.5	50-114
PCB-1221	50	24.4	22.1-75.2	15-178
PCB-1232	50	17.9	14.0-98.5	10-215
PCB-1242	50	12.2	24.8-69.6	39-150
PCB-1248	50	15.9	29.0-70.2	38-158
PCB-1254	50	13.8	22.2-57.9	29-131
PCB-1260	50	10.4	18.7-54.9	8-127

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4 of Method 608).

X = Average recovery for four recovery measurements, in µg/L (Section 8.2.4 of Method 608).

P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2 of Method 608).

D = Detected; result must be greater than zero.

Note: These criteria are based directly upon the method performance data in Table 8 of Method 608. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 8.

Table 8
Method Accuracy and Precision as Functions of Concentration—Method 608

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
PCB-1016	0.81C+0.50	0.13X+0.15	0.15X+0.45
PCB-1221	0.96C+0.65	0.29X−0.76	0.35X−0.62
PCB-1232	0.91C+10.79	0.21X−1.93	0.31X+3.50
PCB-1242	0.93C+0.70	0.11X+1.40	0.21X+1.52
PCB-1248	0.97C+1.06	0.17X+0.41	0.25X−0.37
PCB-1254	0.76C+2.07	0.15X+1.66	0.17X+3.62
PCB-1260	0.66C+3.76	0.22X−2.37	0.39X−4.86

X′ = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration found of X, in µg/L.

S′ = Expected interlaboratory standard deviation of measurements at an average concentration found of X, in µg/L.

C = True value for the concentration, in µg/L.

Table 9
Summary of Method 608 QC Requirements

QC Objective	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Prior to sample analysis; as needed thereafter when CCV fails or major maintenance; column change	<10% RSD to use average RF; Regression curve if >10% RSD	Re-prep and re-curve
Calibration Curve Verification (CCV)	Each work day prior to sample analysis	± 15% of the expected response	Repeat once using a fresh calibration standard; New calibration curve.
Matrix Spike (MS)	10% of the samples from each sample site	Compare recovery (P) with acceptance criteria in Table 7	Analyze QC Check Standard (QCCS) and compare recovery (Ps) with acceptance criteria in Table 7.
Quality Control Check Standard (QCCS)	Analyzed if any analyte in matrix spike is outside QC acceptance limit.	Compare recovery (Ps) with acceptance criteria in Table 7	Recovery for the QCCS is evaluated for any parameter that is outside the QC acceptance criteria in the matrix spike (MS). I fa parameter is outside acceptance criteria in both the MS and QCCS, that parameter can not be reported for regulatory purposes.

Title: Gas Chromatographic Analysis of Total Petroleum Hydrocarbons [Methods AK 102/103, SW846-8015B, SW846-8015D, CA-LUFT]

Approvals (Signature/Date):

11/12/14	11-12-2014
Victoria Nihart — Date	Joe Schairer — Date
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11/13/2014	11/17/14
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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures to be used for determination and/or quantitation of semivolatile hydrocarbon mixtures that elute between n-C8 (octane) and n-C36 (hexatriacontane). Extension of the chromatographic run to include to n-C40 (tetracontane) may be performed as a client-specific measure. This SOP is applicable to extracts that are prepared according to the appropriate sample extraction SOPs (WS-OP-0004).

1.2. Table 1 lists hydrocarbon fuel mixtures that are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs are based on the low level standard and the sample preparation concentration factors. Matrix interferences or limited sample volume may result in higher RLs than those listed.

1.3. This method is designed to measure mid-range petroleum products such as Diesel #2 or motor oil utilizing methods AK102/103, SW846-8015B and SW846-8015D. As of the September 2012 release of the leaking Underground Fuel Tank Guidance Manual, CA_LUFT samples are to be run per SW846-8015B protocols and criteria.

1.4. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts for hydrocarbon fuel mixtures. The extracts are injected onto the column and separated and detected by flame ionization detection. Quantitation is by the external standard method.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID).

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the flame ionization detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Other organic compounds including animal and vegetable oil and grease, chlorinated hydrocarbons, phenols and phthalate esters are measurable under the conditions of this method. As defined in the method, the unknown hydrocarbon results include these compounds.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including silica gel cleanup. A silica gel cleanup can remove most of the polar organic interferences. These cleanup procedures are included in SOP WS-OP-0004.

4.5. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for carryover.

4.6. Petroleum products are complex mixtures of compounds derived from crude petroleum, and different products may have overlapping boiling ranges and chromatograms. The products are also subject to degradation in the environment with consequent changes in the chromatographic profile. When such changes are considerable, it may not be possible to positively identify the parent mixture.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when working with solvent or extract containers. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.2. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable. All preparation of standards and dilutions shall be performed inside an operating fume hood. All samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

6.1. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

GAS CHROMATOGRAPH(1)	Replace septum. Clean injector port Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace igniter. ECD: follow manufacturers suggested maintenance schedule PID: Clean lamp window or replace. Replace seals. Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.	As Needed
	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily(2)
	Oxidation and Reduction Catalysts: Perform leak checks. Replace/condition when poor response is observed.	Quarterly
	ECD: perform wipe test.	Semi-Annually

6.2. Gas Chromatograph - Agilent model 6890N or equivalent.

6.2.1. Autosampler – Agilent model 7673, 7683B or equivalent.

6.2.2. Operating System – Agilent MSD ChemStation D.02-00-275 or equivalent.

6.2.3. Software version –Chrom Peak Review, Version 2.1. or equivalent.

6.3. Preventive and routine maintenance is described Section 20.2 of the Sacramento Quality Assurance Manual, and in Table 9 of this SOP.

6.4. Refer to Table 3 for analytical column and conditions.

6.5. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Neat Standard Materials

Hydrocarbon fuels are generally available as stock solutions from commercial vendors. In rare instances when they are not available from vendors, neat materials may be acquired directly from the client. Such neat materials expire following a period of 3 years from the date of receipt, or sooner if problems such as pattern degradation or linearity loss occur.

7.2. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Standard solutions are stored at 0 - 6°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Stock standard solutions must be replaced after six months (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Stocks may be replaced sooner if comparison with check standards prepared from an independent source indicates a problem.

7.3. Calibration Standards

Calibration standards are prepared as dilutions of the stock standards. Surrogate standards are used as specified. Calibration solutions must be refrigerated at 0 - 6°C and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated or if comparison with check standards indicates a problem.

Refer to Table 3 for details of calibration standards.

7.4. Surrogate Standards

7.4.1. Ortho-terphenyl is the surrogate standard. Refer to Tables 4 and 6 for details of the surrogate standard.

7.4.2. The surrogate n-triacontane-d62 is required for most AFCEE projects and may be used as a surrogate for AK103 (RRO) analysis. Refer to Tables 4 and 6 for details of this surrogate standard.

7.5. Alkane Range

A single level standard containing the even-numbered alkanes from n-C8 to n-C40, n-C19 and n-C25, is used to set retention time windows for fuel identification and

quantitation. This may be made from multiple commercially available standards. The alkane range solution has an expiration of one year from date of preparation or the manufacturer's expiration of the solution(s) from which it is diluted, whichever is earlier. Refer to Table 5 for details of the alkane range standard.

7.6. Gases for carrier, make-up, and detector fuel: hydrogen, and nitrogen, and air.

7.7. Quality Control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They may be made from the same stock as the calibration standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Semivolatile extracts must be refrigerated at 0 - 6°C. Aqueous samples must be extracted within 7 days of collection and solid samples within 14 days of collection. Extracts must be analyzed within 40 days.

8.2. Aqueous samples for analysis by AK102 must be preserved to pH < 2 and extracted within 14 days of collection. Solid samples for both AK methods must be extracted within 14 days of collection. All samples for both AK methods must be analyzed within 40 days of collection (not extraction).

NOTE: Holding times for AK methods are calculated to the hour.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is the analysis of a single point calibration standard.

9.2. Quality Control Batch

The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch

may contain multiple MS/MSDs. See policy WS-PQA-003 for further definition of the batch.

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC Program document (WS-PQA-003) for further details of the batch definition.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). Refer to policy WS-PQA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction). Surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into TALS or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (WS-PQA-003) for further details of control limits.

9.3.4. Refer to Table 10 for AK 102/103 recovery requirements for surrogates and laboratory control samples (LCS/LCSD) and RPD (Relative Percent Deviation).

9.4. Surrogates

Surrogate recoveries in field samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Reprepare and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem. Repreparation is not necessary if there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with QA

and the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.4.2. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.5. Method Blanks

One method blank must be processed with each preparation batch. The method blank consists of reagent water for aqueous samples. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher. Certain programs, such as DOD QSM, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.

9.5.1. Re-preparation and reanalysis of any samples with reportable concentrations of analytes less that 10 times the value found in the method blank is required unless other actions are agreed with the client.

9.5.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. This must be documented in the NCM program.

9.5.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.5.4. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.6. Laboratory Control Samples (LCS)

9.6.1. For each batch of samples, analyze an LCS. The LCS contains a

representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur.

9.6.1.1. LCS compound lists are included in Table 6.

9.6.1.2. For AK Methods (AK102; AK103) prepare and analyze an LCS Duplicate sample as well as an LCS sample for each component of interest (DRO, RRO) for each analytical batch.

9.6.2. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:

- Check calculations,
- Check instrument performance,
- Reanalyze the LCS, and if still outside of control limits,
- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.6.3. With the exception of QSM 5.0 projects, data may be reported with an anomaly in the following cases:

- The LCS recoveries are high and the analyte of concern is not detected in field samples,
- All requested target analytes are within control, but other LCS compounds are out of control.

9.6.4. The analyst should evaluate the anomalous analyte recovery for possible trends.

9.6.5. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

9.6.6. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.7. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.7. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 6. Compare the percent recovery and relative

percent difference (RPD) to those in the laboratory specific historically generated limits.

9.7.1. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The first corrective action is to evaluate the MS/MSD for any systemic issues such as mislabeling, incorrect spiking, bad injection, etc. If the failure is due to these issues it must be re-injected or re-extracted depending on the situation. If no systemic problem is found corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.

9.7.2. If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

9.7.3. If a MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed if desired by the client.

9.7.4. The MS/MSD must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.7.5. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. If TALS has appropriately flagged the data, an NCM is not necessary. Otherwise, file an NCM stating that the 4X rule was applied.

9.8. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of an LCS pair in place of an MS/MSD must be documented using the NCM module in TALS.

9.9. Initial Calibration Verification (ICV) -- An ICV must be run after all points to be used in the curve have been run and before any samples are run. The ICV is from a certified second source and should be at a concentration roughly at the midpoint of the calibration. All analytes to be reported must be in the ICV.

Each compound of the second source calibration must be within the criteria below when evaluated with respect to the calibration curve:

Method/Program	%D from expected value
Method 8000B/8015B	± 15%

Method 8000C/8015D	± 20%
DoD QSM	± 20%
AK102/103	± 25%
CALUFT	± 20%

Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards

9.10. Certain clients may require specific project or program QC which may supersede these method requirements. The analyst should check the project notes for the job in TALS before running samples.

9.11. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager.

9.12. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. Initial Calibration

10.1.1. External calibration is used. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Table 4.

10.1.2. The alkane range standard should be run with the initial calibration. If the initial calibration consists only of add-on mixtures, and the current diesel fuel calibration standards indicate that no peak shifting has been occurring, analysis of the alkane standard may be waived.

10.1.3. Quantitation of hydrocarbon mixtures

10.1.3.1. Starting and ending retention times for quantitation are determined for each fuel. The peak areas between the starting and ending times are summed and used to generate a response factor. This factor is used to quantitate sample results, and depending on the client

requirements, this factor may be applied to the same retention range as the standard, or to a different range.

10.1.3.2. See Table 2 for details of the commonly used retention and quantitation ranges.

10.1.3.3. When setting starting and ending retention times based on Table 2 ranges (or other commonly used ranges), the start of the range should be set ± 0.1 minute before the first marker peak and end ± 0.1 minute before the last marker peak. The retention times of the apexes of the marker peaks should not be used as the retention time windows for the fuels.

10.1.4. Analyze an ICV (Section 9.9) immediately following the calibration curve and before field sample analysis. This verification standard is used as a quality control check to verify the accuracy of the calibration.

10.2. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, or changing PID lamps or FID jets, and any changes in instrument operating parameters, including gas flows, detector temperatures, oven temperatures, etc.

10.3. With the exception of instances detailed in Policy CA-T-P-002, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve.

10.4. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

Equation 1

$$Calibration\ Factor\ (CF) = \frac{Area\ \ of\ \ Peak(s)}{Concentration\ of\ \ Standard\ (ng/ml\ or\ ug/ml)}$$

Due to the nature of hydrocarbon fuels, peak height may not be used for quantitation. Variations on this formula are permitted so long as the calculations between standards and samples remain consistent.

10.5. Average response factor or linear regression may be used to fit the data. Linear regression may be used if the calibration range is sufficiently narrow so that a straight line will fit the calibration points. Average response factor may be used if the %RSD of the response factors or calibration factors is ≤ 20%.

10.5.1. Average response factor

10.5.1.1. The average response factor may be used if the percent relative standard deviation (%RSD) of the response factors is ≤ 20%.

10.5.1.2. The equation for average response factor is:

Equation 2

$$\text{Average response factor} = \frac{\sum RF_{1-n}}{n}$$

Where: n = Number of calibration levels

$\sum RF_{1-n}$ = Sum of response factors for each calibration level

10.5.2. Linear regression

10.5.2.1. The linear fit uses the following function:

Equation 3

$$Concentration = A + BR$$

Where: A = Intercept

B = Slope

R = Response

10.6. Evaluation of calibration curves

10.6.1. The percent relative standard deviation (%RSD), or percent relative standard error (%RSE) for non-average response factor calibration models, from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.6.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level.

10.6.2.1. The percent relative standard error (%RSE) is calculated as follows:

Equation 4

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{Ci - PCi}{Ci}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve

P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)

C_i = True concentration for level i

PC_i = Predicted concentration for level i

Note that when average response factors are used, this equation gives the same value as the %RSD of the response factors.

10.6.2.2. Percent relative standard deviation is calculated as follows:

Equation 5

$$\% RSD = 100\% \times \frac{\sqrt{\frac{\sum_{i=1}^{N}\left[RF_i - \overline{RF}\right]^2}{N-1}}}{\overline{RF}}$$

Where:

N = Number of points in the curve

RF_i = Response factor calculated for level i

$\overline{RF}$ = Average response factor

10.7. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- Relative standard deviation (or RSE) of the calibration points from the curve used must meet the criteria below:

Method/Program	RSD
Method 8000B/8015B	20%
Method 8000C/8015D	20%
DoD QSM	20%
AK102/103	25%
CALUFT	20%

- In some cases, (normalized) %RSD may not be appropriate (i.e., curve fits without 1/Concentration^2 weighting). In this case, the correlation coefficient (r-squared) may be used as an alternative to the %RSD, and must be greater than 0.990.

Note: The normalized %RSD is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have very good correlation coefficient (>0.995), while also having >100% error at the low point.

10.7.1. Weighting of data points

In a linear calibration fit, the points at the lower end of the calibration have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

NOTE: Prior to accepting an initial calibration curve, it is highly recommended to recalculate the calibration levels using the new calibration response factors. Then calculate the % difference for every analyte and every level of the calibration and compare to the criterion of ≤20% from the true concentration. If the curve fails this criteria consult the department manager about whether to re-do the calibration.

10.7.2. Non-standard analytes are sometime requested. For these analytes, it is acceptable to analyze a single standard for pattern identification daily, rather than a five point initial calibration, provided that the client agrees with this strategy.

10.7.2.1. Positives which match the pattern of the non-standard analyte may be quantitated based on a standard reference if the elution range is similar and the client agrees to this. In this instance, "unknown hydrocarbons" are quantitated based on the default material for the fraction (e.g., diesel, motor oil, or gasoline, based on elution range).

10.7.2.2. Otherwise, a five-point initial calibration must be run and samples with positives must be reanalyzed for quantitation. If the analyte is not detected, the non-detect may be reported and no further action is necessary.

10.8. Integration

10.8.1. When evaluating the initial calibration, also evaluate hydrocarbon pattern integration.

10.8.1.1. The integration baseline should run from the point in the chromatogram prior to peaks to a region following the mixture's peaks in a fairly straight line.

10.8.1.2. If there is unresolved material in the middle of the chromatogram, the area under the "hump" should also be integrated.

10.8.2. The default integration parameters generated as a result of evaluating the initial calibration should remain in effect until the initial calibration is reanalyzed.

10.9. Calibration Verification (CCV)

10.9.1. The working calibration curve or RF must be verified by the analysis of a mid-point calibration standard. For Method 8015B and 8015D the CCV must be run at the beginning, after every 20 field samples or 12 hours, whichever is more frequent, and at the end of the analysis sequence. Specific details may be found in method 8000C section 11 and paragraph 9.2.2. For QSM 5.0 work the CCV must be run at the beginning, after every 10 field samples or 12 hours, whichever is more frequent, and at the end of the analytical sequence.

10.9.2. The center of each retention time window for surrogate compounds is updated with each CCV. For fuel standards, the fuel's elution profile is compared to the initial calibration to verify that shifting has not occurred since initial calibration. If an alkane range standard has been analyzed, it is checked to verify that shifting has not occurred since the retention time windows for the fuels were set.

10.9.3. It may be appropriate to analyze a mid-point standard more frequently than every 20 samples (or 12 hours, whichever is more frequent). If these continuing calibration standards are analyzed, requirements are the same.

10.9.4. Each compound within the CCV must be within the criteria below when evaluated with respect to the calibration curve.

Method/Program	%D from expected value
Method 8000B/8015B	± 15%
Method 8000C/8015D	± 20%
DoD QSM	± 20%
AK102/103	± 25%
CALUFT	± 20%

If these criteria are exceeded, then corrective actions must be implemented. Typically, samples analyzed after the nonconforming CCV are reanalyzed. Samples analyzed prior to the nonconforming CCV are also reanalyzed, unless the samples are non-detect and the CCV is exhibiting an elevated response. A non-conformance memo must be filed in this instance. Refer also to Section 10.9.6

NOTE: Samples analyzed under DOD QSM version 5 must have passing CCVs both before and after them.

10.9.4.1. % Difference calculation

Equation 6

$$\% \text{ Difference} = \frac{|\text{Expected value} - \text{Calculated value}|}{\text{Expected value}} \times 100\%$$

10.9.5. It is not necessary to run a continuing calibration standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.9.6. If the analyst notes that a CCV has failed and can document the reason for failure (e. g. no purge, broken vial, carryover from previous samples, etc) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV then the preceding samples have not been successfully bracketed but analysis may continue. For corrective actions required for QSM projects see WS-PQA-021.

10.9.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples.

10.9.8. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.9.9. CCV analysis for Methods AK102.0 and AK103.0 (State of Alaska) analysis includes analysis of the alkane range standard to verify retention times.

Certain programs, such as those falling under the DOD QSM, require that the alkane range standard be run daily to verify retention times.

10.10. Refer to Table 3 for details of GC operating conditions.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Extraction

The extraction procedure is described in SOP WS-OP-0004.

11.3. Cleanup

Cleanup procedures are described in WS-OP-0004.

11.4. Suggested gas chromatographic conditions are given in Table 3. Actual gas chromatographic conditions can be found in the maintenance logs.

11.5. Instrument Bleed Profile

Because the extractable hydrocarbon analysis is subject to integration difficulties as a result of column bleed and late-eluting compounds, it is advisable to subtract the instrument bleed profile from every injection. Profiles should be renewed when there is an obvious change in the instrument's column bleed profile. Only instrument runs performed without injection of solvent and where the chromatogram shows no obvious peaks may be used for bleed profiles.

11.6. Analytical Sequence

An analytical sequence starts with an initial calibration or a calibration verification (CCV), performed as described in Sections 10.1 and 10.9.

11.6.1. If there is a break in the analytical sequence of greater than 2 hours, the sequence must be restarted with a DCM blank.

11.7. Retention Time Windows - Single Peaks

11.7.1. Retention time windows must be determined for all single peak analytes (including surrogate compounds). Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.7.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows for surrogate compounds are updated with the mid-point of the initial calibration and each daily calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.7.3. If the retention time window as calculated above is less than ± 0.03 minutes,

use ± 0.03 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.7.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.7.5. The retention times of all surrogate compounds in each continuing calibration must be within the retention time windows established by the daily calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

11.8. Retention Time Windows - Multiple Peak Standards

Because multiple peak hydrocarbon mixtures are quantitated based on the total area between two retention times, the retention time windows that are generated are also used to determine the times used for area summing.

Retention time windows must be determined for each hydrocarbon mixture to be analyzed.

11.8.1. Make an injection of the alkane range standards once per day over a three day (72 hour) period. Calculate and tabulate the standard deviation of the three retention times for each peak in the mixture. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.8.2. Hydrocarbon mixture summing windows are the first peak minus three times the standard deviation for that peak to the last peak plus three times the standard deviation for that peak. For example, for a fuel quantitated between n-C10 and n-C24, the peak summing window is from the retention time for n-C10 minus three times the standard deviation for n-C10 to the retention time for n-C24 plus three times the standard deviation for n-C24.

11.8.3. For mixtures which use an alkane which is not present in the carbon range reference, the retention time of the alkane may be determined by linear extrapolation, and the retention time window of the alkane following the one not present may be used to set peak summing windows. An example of this instance is JP-4 in the extractable hydrocarbons test, which uses n-C13 as the end of the summing window.

11.8.3.1. Linear Interpolation of Retention Times

Equation 7

$$RT_{Int} = RT_{Earlier} + \frac{(RT_{Later} - RT_{Earlier})}{2}$$

Where:

RT_{Int}	=	Interpolated Retention Time (for n-C13 in the example above)
$RT_{Earlier}$	=	Retention Time of earlier peak (for n-C12 in the example above)
RT_{Later}	=	Retention Time of later peak (for n-C14 in the example above)

11.8.4. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows for the alkane range peaks are updated with the alkane range that is run with new initial calibrations. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.8.4.1. Retention time windows for hydrocarbon mixtures are updated at the same time as those for the alkane peaks.

11.8.4.2. If an alkane range is not run with the initial calibration, retention times and summing windows may not be updated.

11.8.5. If the retention time window for an alkane peak as calculated above is less than ± 0.03 minutes, use ± 0.03 minutes as the retention time window.

11.8.6. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Because quantitation of hydrocarbon mixtures is dependent on the peak summing window, calibration data evaluation and sample analysis cannot continue until the alkane retention time windows are established.

11.8.7. Because quantitation of fuels is based on the total area between two set retention times, each fuel standard's elution profile (peak pattern with respect to retention times denoting start and end of quantitation) in each continuing calibration must be similar to the profile at the time of initial calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

11.8.7.1. Compare elution profiles by utilizing the data system's capability to overlay chromatograms on-screen, and overlay the mid-level standard from the calibration curve with the daily calibration.

11.8.7.2. Alternatively, compare elution profiles by overlaying hard-copy chromatograms that are at the same scale, and comparing them.

11.8.7.3. Certain methods may require that the alkane standard be run prior to each analytical batch, and that retention times for this standard are evaluated. Such instances are detailed in the appendices.

Note: When exaggerated shifting of surrogate compounds occurs in samples, quantitation of any fuel mixture, unknown or otherwise, is likely to be adversely affected. Retention time shifting can be a result of instrument gas leaks, poor temperature control at the instrument, poor laboratory temperature control, or matrix effects. It is highly recommended that the cause is determined and samples reanalyzed when retention time shifting occurs.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Identification

Tentative identification occurs when a peak pattern matching the reference standard is found within the elution window for a fuel, at a concentration above the reporting limit, or above the MDL if J flags are required. If the peaks present in the chromatogram elute within the time window of the reference fuel, but the pattern does not match the reference fuel, then the material is determined to be an "Unknown Hydrocarbon" and will be reported accordingly.

12.1.1. "J" values for Unknown Hydrocarbons

Typically, unknown hydrocarbons are not reported as "J" values, or values below the reporting limit. This is because "Unknown Hydrocarbons" is a category which can both describe fuels too degraded to identify, as well as biologic by-products and other contaminants. Any quantitation of an unknown hydrocarbon is an estimate, as no true reference exists.

12.1.2. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

Note: In cases of extremely oily samples, it may be advisable to dilute samples such that the analyte concentration is in the lower half of the calibration range. Such deviations should be noted and filed with the project.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the peaks in the Level 3 standard, then the sample should be reanalyzed at a more concentrated dilution.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.5.1. Aqueous Samples (assuming average response factor calibration)

Equation 8

$$Concentration\ (\mu g / L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_s)}$$

Where:

A_x = Response for the analyte in the sample

D_f = Dilution factor

V_t = Volume of total extract, mL

V_s = Volume of sample extracted or purged, mL

CF = Calibration factor, area/ng/mL or area/µg/mL, Section 10.1

12.5.2. Non-aqueous Samples (assuming average response factor calibration)

Equation 9

$$\text{Concentration}(\mu g / kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\text{Moisture}}{100}$$ (D = 1 if wet weight is required)

Note: The percent moisture correction may occur following entry into a LIMS database instead of at the instrument.

A_x, V_t, D_f, and CF are as defined in Equation 8

12.6. Identification of Multi-component Analytes

12.6.1. Chromatograms are evaluated only for the fuel list specified by the client. "Fingerprinting" of materials is not normally performed beyond the specified list.

12.6.2. If the material present in the client sample does not elute in a range similar to the reference(s), and the chromatographic profile is not the same, then the material is reported as an "Unknown Hydrocarbon" or similar generic title.

12.7. Evaluating Degraded Diesel #2

12.7.1. All fuels exposed to the environment are subject to a variety of weathering and degradation agents, and therefore they may not be identified after a period of time in the environment. To aid in the identification of degraded Diesel #2, four criteria should be evaluated. If at least two of the four criteria are met, but the profile is not a good match for the diesel standard, then it can be considered a degraded diesel fuel and reported as diesel fuel, with a footnote denoting that it is degraded.

12.7.2. Criteria for evaluating degraded Diesel Fuel #2

- The OTP peak (about C-19) is distinctly on the downslope of the unresolved material.
- All alkane peaks from n-C10 to n-C23 are visible.
- The envelope of unresolved material returns to the baseline between n-C23 and n-C26.
- The baseline rise for the unresolved material starts by n-C11 and the early end from n-C10 to n-C13 is present.

12.8. Surrogate recovery results are calculated and reported for Ortho-terphenyl (OTP). When this analysis is performed in support of method AK103, results for n-triacontane-d62 must also be reported. In some client specific cases, other surrogate compounds may also be used. Due to the nature of hydrocarbon fuel analysis, surrogate recovery may be elevated beyond the upper control limit as a result of area contribution from a fuel analyte. In these instances, report the recovery, and narrate the elevated recovery in the case narrative.

12.9. When reporting Diesel Range Organics (DRO), no Unknown Hydrocarbon is evaluated. If there is a concentration above the reporting limit in the DRO range, then there is DRO present.

12.10. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

Equation 10

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

12.11. Reporting Requirements

12.11.1. Results are reported as described in the method appendices.

12.11.2. Reporting limits are listed in the method specific appendices.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the

method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits referenced in Table 7 (Performance limits, four replicate initial demonstration of capability).

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials contaminated with methylene chloride. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After the autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methylene chloride to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996. Section 8000B

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV, March 2003. Section 8000C

16.3. Leaking Underground Fuel Tank Guidance Manual, State of California, September, 2012

16.4. Underground Storage Tanks Procedures Manual, Alaska Department of Environmental Conservation, November 7, 2002; Appendix D

16.5. Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997

16.6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Edition, Final Update III, December, 1996; Method 8015B

16.7. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Edition, Final Update IV, March 2003; Method 8015D (Revision 4, June 2003)

16.8. Alaska Department of Environmental Conservation, April 8, 2002; Methods AK102.0 and AK103.0

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. ADEC methodology specifies that standards and extracts are stored at –10 to –20°C. This SOP used 4 ± 2°C as the requirement. This temperature range is consistent with other SOPs at this facility.

18. ATTACHMENTS

18.1. Table 1 – Standard Analyte List and Reporting Limits

18.2. Table 2 – Extractable Petroleum Hydrocarbon Quantitation Ranges

18.3. Table 3 – Parameters and Recommended Conditions

18.4. Table 4 – Calibration Levels µg/mL

18.5. Table 5 – Components of Alkane Range Standard

18.6. Table 6 – LCS/Matrix Spike and Surrogate Spike Levels µg/L or mg/kg

18.7. Table 7 – Performance Limits, Four Replicate Initial Demonstration of Capability

18.8. Table 8 – AK102/103 Recovery Requirements

18.9. Table 9 – Recommended Preventative Maintenance

19. REVISION HISTORY

19.1. WS-GC-0007, Revision 5.9, Effective 11/21/2014

19.1.1. Removed Section 10.9.3 – "The mid-level calibration standard used at the start of the analysis sequence must be a different concentration than the mid-level calibration standard used for subsequent calibration verifications. Recommended concentrations are noted in Table 4."

19.1.2. Editorial changes.

19.2. WS-GC-0007, Revision 5.8, Effective 06/27/2014

19.2.1. Deleted Section 7.4.3 – "Samples to be run in accordance with CA LUFT methodology are spiked with capric acid as a reverse surrogate added prior to silica gel cleanups. Detection of this surrogate in the final extract indicates the presence of water and/or an overloaded cleanup column" as this procedure is not being used at TestAmerica Sacramento.

19.2.2. Revised Section 7.7 - "QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They may be made from the same stock as the calibration standards.

19.2.3. Editorial changes.

19.3. WS-GC-0007, Revision 5.7, Effective 10/21/2013

19.3.1. Removed all references to NWTPH methods as they are no longer offered by the laboratory. This includes edits to the Title, Section 1.3, and removal of Section 8.2, Section 12.10, 16.9 and 17.5.

19.3.2. Updated to reflect the current data processing and LIMs system, which permits easy use of multiple program acceptance criteria for QC elements. Consequently, Sections 17.5 and 17.6 have been removed, as the less stringent AK criteria have been programmed into the data processing system for use with AK samples.

19.3.3. Replaced/Updated CA-LUFT references to reflect the September 2012 manual. Section 17.2 and 17.3 have been removed, Section 16.3 has been revised.

19.3.4. Section 4.4, added, “A silica gel cleanup can remove most of the polar organic interferences.” in the middle of the paragraph.

19.3.5. Section 6, updated the data processing software to the current Chrom software, and changed the reference for preventative maintenance to Table 9 of this SOP.

19.3.6. Added Section 7.4.3 (Capric acid reverse surrogate).

19.3.7. Section 7.5 and Table 5, added n-C19.

19.3.8. Added Table 9

19.3.9. Changed all references to QuantIMS to TALS.

19.3.10. Added corrective actions to Section 9.7.1.

19.3.11. Removed Section 12.11.3, as the information is now all stored and available in the LIMS and data processing software to the data reviewer.

19.3.12. Updated Section 9.9 to include criteria for all method variations covered by this SOP, as well as to clarify when the ICV must be analyzed.

19.3.13. Updated Section 10.7 and 10.9.5 criteria, added the note following Section 10.7.1, and updated Section 10.9.1 frequency.

19.3.14. Added references to SW-846 Update IV methods (8000C, 8015D).

19.3.15. Editorial revisions

19.4. WS-GC-0007, Revision 5.6, Effective 05/31/2013

19.4.1. Changed Clouseau references to ’NCM module in TALS’.

19.4.2. Replaced footer in Table I to reference reduced volume extractions.

19.4.3. Removed footnotes following Table 4.

19.4.4. Editorial changes.

19.5. WS-GC-0007, Revision 5.5, Effective 09/14/2012

19.5.1. Removed Table 8 and Table 9 due to TALS implantation.

19.5.2. Remove Section 12.8 “Reporting Unknown Materials as this descriptor is not used with TALS/Chrom reporting.

19.5.3. Editorial changes

19.5.4. Editorial revisions

Table 1 Standard Analyte list and Reporting Limits			
Fuel	Quantitation Range	Reporting Limit, µg/L or mg/kg	
		water	soil
Diesel Fuel #2	n-C10 to n-C24[ERS1]	100	5
Additional Fuels			
Motor Oil (10W-40)	n-C19 to n-C36	500	10
JP-4	n-C8 to n-C13	250	5

	Sample Vol.	Final Vol.
Water	1000 mL	3 mL
Reduced Volume Extraction (LVI/RVE)	250 mL	2 mL
Soil/Solid	30 g	3 mL
Low Level Soil/Solid by EPA 3586 (MAE)	15 g	2 mL

Table 2 Extractable Petroleum Hydrocarbon Quantitation Ranges.			
Regulatory Method	Component	Quantitation Range	
EPA 8015B EPA 8015D	Diesel Fuel	n-C10	n-C28
	Mineral Oil	n-C15	n-C40
	Mineral Spirits	n-C8	n-C13
	JP-4	n-C8	n-C13
	JP-5	n-C8	n-C16
	JP-8	n-C8	n-C18
	Hydraulic Oil	n-C20	n-C40
	Kerosene	n-C8	n-C16
	Fuel Oil #6	n-C10	n-C16
	Stoddard Solvent	n-C8	n-C13
CA-LUFT (Default)	Diesel Range Organics (DRO) Oil Range Organics (ORO)	n-C12 n-C23	n-C22 n-C32
AK102.0[1] AK103.0[2]	DRO RRO	n-C10 n-C25	n-C25 n-C36

[1] *The method specifies that the peak summing window must start at the start of the n-C10 peak and end at the start of the n-C25 peak.*

[2] *The method specifies that the peak summing window must start at the start of the n-C25 peak and end at the start of the n-C36 peak.*

Table 3	
Parameter	**Recommended Conditions**
Injection port temp	250°C
Detector temp (FID)	320°C
Injection	2µL
Injection Type/Splitter	Splitless, 0.3 min purge time, 20 mL/min purge flow
Column	DB-5.625, 30m x 0.32 mm, 0.25 µm (or equivalent)
Temperature program	50°C for 2.0 min, 15°C/min to 320°C, 3 min hold
Flow Program	5 mL/min for 2.0 min, 2 mL/min to 12 mL/min, hold to end of run
Gas Saver	On, flow = 20 mL/min
Make up gas flow mode	Makeup + column flow = constant, set for 50 mL/min
Carrier Gas	Hydrogen
Make-up Gas	Nitrogen
Fuel Gas	Hydrogen, 40 mL/min
Oxidant Gas	Air, 450 mL/min

Table 4							
Calibration Levels µg/mL							
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**
Diesel Fuel	10	50	100	300	500	1000	2000
Motor Oil (10W - 40)	50	100	300	500	800	1200	1500
JP-4	10	50	100	250	500	1000	
o-terphenyl (surrogate)	1	5	10	15	20	25	
n-triacontane-d62 (surrogate)	1	5	10	15	20	25	

Table 5	
Components of Alkane Range Standard[1]	
Component	**Approximate Concentration (µg/mL)**
n-C10 (decane)	5
n-C12 (dodecane)	5
n-C14 (tetradecane)	5
n-C16 (hexadecane)	5
n-C18 (octadecane)	5
n-C19 (nonadecane)	5
n-C20 (cosane)	5
n-C22 (docosane)	5
n-C24 (tetracosane)	5
n-C25 (pentacosane)	5
n-C26 (hexacosane)	5
n-C28 (octacosane)	5
n-C30 (triacontane)	5
n-C32 (dotriacontane)	5
n-C34 (tetratriacontane)	5
n-C36 (hexatriacontane)	5
n-C38 (octatriacontane)	5
n-C40 (tetracontane)	5

[1] Standard is used only for setting retention times; therefore, exact concentrations are not necessary. A concentrated mixture of n-alkanes from n-C10 through n-C40 is available from Ultra Scientific. Its use is recommended.

Table 6		
LCS/Matrix Spike and Surrogate Spike levels µg/L or mg/kg		
	Aqueous	**Soil**
Diesel Fuel	300	10
o-Terphenyl (Surrogate)	40	1.33
Motor Oil	900	30
n-Triacontane-d32	50	1.67

Table 7		
Performance limits, four replicate initial demonstration of capability		
Compound	**Initial demonstration, mean recovery limits**	**Initial demonstration, RSD limits**
Diesel Fuel	50-150	25
Motor Oil (10W - 40)	50-150	25
JP-4	50-150	25
Other Add-on Fuels	50-150	25

Table 8	
AK102/103 Recovery Requirements	
Requirement	**Criteria**
Surrogate recovery in samples	50-150%
Surrogate recovery in CCV	60-120%
Surrogate recovery in LCS/D	60-120%
LCS Recovery	75-120% (Diesel); 60-120% (AK-103)
LCS/LCSD RPD	Less than or equal to 20%

Table 9	
Recommended Preventative Maintenance for GC-FID	
Maintenance	**Frequency**
Replace septum. Clean injector port Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace igniter. Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.	As Needed
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily (when in use)

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Title: Determination of Fixed Gases (Reformed Gases) in Air Samples using Gas Chromatography

[Methods ASTM D-1946 / EPA 3C / ASTM D-1945 / ASTM D-3588]

Approvals (Signature/Date):

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Lisa Stafford — 7/21/2014 — Date Quality Assurance Manager	Robert Hrabak — 7/21/14 — Date Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the gas chromatographic analysis of air or vapor samples for fixed gas compounds using thermal conductivity detectors (Front TCD/Aux TCD).

1.2. This SOP is restricted for use by experienced analysts or those under the supervision of analysts experienced in sample preparation, use of gas chromatography (GC), and interpretation of chromatograms.

1.3. Analytes, Matrix, and Reporting Limits (RLs)

1.3.1. Target analytes and RLs are listed in Attachment 1. Note that RLs are subject to change based on annual LOQ/LOD studies.

1.3.2. Applicable matrices – ambient air, indoor air

1.4. This SOP also described the calculations used to report Natural Gas composition (ASTM D1945) and "Heat Value and Relative Density of Gaseous Fuels" (ASTM D-3588). To calculate these values, results from Method TO-3 (WS-GCA-0018) are also required.

1.5. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. An air or vapor sample is injected with a gas-tight syringe into a sample loop or by directly connecting a canister to the sample line and onto HaySep and Mol Sieve columns. The system automatically adjusts the sample loop to one atmosphere before injection. Oxygen(O2), nitrogen (N2), and methane (CH4) are the first to pass through the HaySep and are then directed onto the Mol Sieve. After they have eluted onto the Mol Sieve, the Mol Sieve is isolated. Carbon dioxide (CO2) is next to elute from the HaySep, bypassing the isolated Mol Sieve, then goes to the TCD. Carbon monoxide (CO) is last to elute from the HaySep, bypassing the isolated Mol Sieve, then goes to the TCD. Ultra high purity (UHP) helium (He) is used as the carrier gas.

2.2. Hydrogen (H2) and Helium (He) may also be analyzed. UHP N2 is used as the carrier gas. After the elution of these compounds from the HaySep onto the Mol Sieve, the HaySep is back-flushed to vent. H2 and He elute from the Mol Sieve and are detected by the TCD.

3. DEFINITIONS

3.1. Note that "must" and "shall" in this SOP denote required activities.

3.2. Air Sample Bag: Commonly referred to as FlexFilm or Tedlar bag, in 1.0-L or 3.0-L volume, that is constructed of proprietary material (e.g., SKC or ESS).

3.3. Fixed gases: Fixed gases are a group of atmospheric gases which include N2, O2, CO2, CO, CH4, H2, and He.

3.4. Part per million volume to volume (ppmv or ppm v/v): Concentration expressed as part of gaseous (vapor) volume of pure target compound contained in a million part of gaseous volume of sample. One ppmv is equal to 1/10000 of a % v/v.

Note: This reporting unit is NOT equivalent to the common ppm unit used in soil or water analysis.

3.5. Passivated canister: Commonly referred to as SUMMA canister, SilcoCan, or T.O.-Can in 1.0-liter, 1.8-liter, 6-liter, or 15-liter volume.

3.5.1. SUMMA canister: A spherical stainless steel container, which interior has been specially treated by a process (SUMMA passivation) that renders all surfaces inert to VOCs.

3.5.2. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3.5.3. T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek Corporation using a proprietary electropolishing process and extensively cleaned using an ultrasonic method that ensures a high-quality passivated surface that maintains the stability of VOCs during storage.

3.6. Percent by volume (% v or % v/v): Concentration expressed as percentage of gaseous (vapor) volume of pure target compound contained in a gaseous volume of sample; also known as mole percent.

3.7. Standard Dry Air: The laboratory bases its analysis/calculations on the following composition:

Nitrogen	78.1%
Oxygen	20.9%
Argon	0.918%
Carbon dioxide	0.034%
% Total	100.000%

3.8. Standard molar volume = 24.45 L/mol at standard conditions (i.e., room temperature of 25°C and standard pressure of 1 atmosphere).

3.9. Standard pressure = 1.0 atmosphere or 14.6 pounds per square inch absolute (psia) or 0 inches of mercury or 0 pounds per square inch gauge (psig), based on laboratory elevation and average barometric pressure.

Note: Full vacuum (0 psia) = -30 inches of mercury vacuum.

3.10. Vacuum/Pressure Gauge: Device used to measure the vacuum or pressure in a passivated canister. Units of measure range from -30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All pressure units are converted to psia.

3.11. Further definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.12. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. The analytical system is relatively free of interferences due to the multiple column configuration and limited number of similar compounds. Compounds of interest are well separated and there are no major baseline upsets to complicate correct peak integration.

4.2. Contamination may occur in the sampling systems if sample containers are not properly cleaned prior to use.

4.3. Air sample bags should also be shown to be free of contamination at levels one-half the RL for all target analytes.

4.4. All other sampling equipment including pumps, flow controllers, and filters must be thoroughly cleaned and checked for leaks to ensure that the filling apparatus will not contaminate samples.

4.5. The potential for result bias due to carry-over exists. When carry-over is suspected, the sample must be re-analyzed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems

associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Pressurized gas equipment is used in this procedure. Be sure all valves and gauges are operating properly and that no equipment is over-pressurized. After changing cylinders, check all gas line connectors for leaks, with soapy water. Release of high pressure gas can cause rapid suffocation.

Danger: H_2 is a flammable high pressure gas. It can form explosive mixtures with air. It may ignite if the valve is opened to air.

5.1.2. When pressurizing canisters, safety glasses must be worn. If pressure from a canister must be released during this process, a face shield must also be worn.

5.1.2.1. Passivated canisters should never be pressurized over 40 psig.

5.1.3. Pressurized gas cylinders must be securely retained. The use of a face shield is required when changing regulators or disconnecting / connecting cylinders..

5.1.4. Air sample bags must not be pressurized, as seam splitting will occur.

5.1.5. Air sample bags may be used for standard preparation. These must be handled with care and must not be over-filled to prevent them from rupturing. Make sure the valves are closed tightly when not in use.

5.1.6. In order to prevent contamination of the laboratory air by the samples, the vent line must be connected to the system outlet and the fume hood must be turned on.

5.1.6.1. The effluents from the sample splitters for the GC must be vented to a fume hood or at a minimum, must pass through a charcoal filter.

5.1.7. The GC oven contains elevated temperature zones. These zones must be cooled prior to an analyst or technician working on the unit. Temperature appropriate gloves must be worn when working with hot or cold items.

5.1.8. Due to high voltage risk, power to the GC must be turned off or disconnected before work can be done on the instrument.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. There are no materials with a health rating of 3 or 4 used in this method **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methane	Flammable at 5% to 15% in air; Asphyxiate	PEL – Simple Asphyxiant	Exposure via inhalation of the gas. Signs and symptoms of acute exposure may include rapid respiration, muscular incoordination, fatigue, nausea, vertigo, unconsciousness, and death.
Helium	Simple Asphyxiate	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular incoordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
Nitrogen	Simple Asphyxiate	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular incoordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
Hydrogen	Flammable, Simple Asphyxiate	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular in-coordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
Carbon Monoxide	Flammable, poisonous, odorless, Asphyxiate.	Simple Asphyxiant	Exposure via inhalation of the gas. Acts on blood causing damage to central nervous system. Can be fatal even with adequate oxygen. Can form explosive mixtures with air. Symptoms of exposure include shortness of breath, headache, confusion, nausea, dizziness, and unconsciousness.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Instrumentation

6.1.1. Agilent 7890A (or similar) GC equipped with a Front TCD and an Aux TCD

6.1.2. Automated data system capable of archiving instrument runs

6.1.2.1. Chemstation is the data acquisition system.

6.1.2.2. Chrom version 2.1 is the data processing system.

6.1.3. Monochrom program for Nitrogen correction

6.2. Supplies

6.2.1. Chromatographic grade stainless steel and/or nickel tubing and chromatographic grade stainless steel connective fittings (Valco, SwageLok, or equivalent)

6.2.2. Stainless steel chromatographic packed columns – HaySep N 1.8 m X 1/8" X 2.0 mm 60/80; Mol Sieve 5A 1.8 m X 1/8" X 2.0 mm 45/60

6.2.3. Assortment of gas-tight syringes from 0.01-mL to 2.0-L volume for standard preparation (Hamilton, SGE, or equivalent)

6.2.4. Pressure regulators for carrier gas, Front TCD/Aux TCD, and standards – single-stage, stainless steel diaphragm

6.2.5. Stainless steel vacuum/pressure gauge capable of measuring from -30 inches of mercury to 30 psig, or transducer and process meter capable of measuring from 0 psia to 50 psia

6.2.6. 7-micron filters (Nupro or equivalent)

6.2.7. Air sample bag, 1- or 3-L (SKC or equivalent)

6.2.8. Passivated canister, 1.0-L, 1.8-L, 6-L or 15-L (SIS, Restek, Anderson Instruments, Rasmussen, or equivalent)

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP He for carrier gas, TCD reference gas, sample preparation, and standard preparation

7.1.2. UHP H_2 for detector combustion and the reduction catalyst

7.1.3. Zero-grade air for detector combustion and the oxidation catalyst

7.1.4. UHP N2 for carrier gas, TCD make-up gas, TCD reference gas, sample preparation, and standard preparation

7.2. Primary Gas Standards

7.2.1. Primary gas standards (CH_4, CO_2, CO, N_2, He, H2, and O_2) are available in various concentrations as mixtures or pure components, and analytically certified by the supplier (Scott-Marrin, Scott Specialty, or equivalent).

7.2.2. Dilutions of primary gas standards are made on a volume/volume basis using serial dilution methodologies. UHP N_2 or UHP He is used as the diluent gas.

7.3. Working gas standards

7.3.1. Working gas standards are prepared in passivated canisters or air sample bags by making dilutions of the primary gas standards. See Attachment 3 for the nominal concentration levels used in the initial calibration (ICAL).

7.3.2. Working Gas Standard Prepared in Canisters - To prepare a working gas standard, an aliquot of the primary gas standard or of the pre-mixed primary gas standard is transferred to a clean and evacuated passivated canister. The aliquot is metered in by measuring the vacuum of the canister as the aliquot is being transferred. The ratio of the final canister pressure and the pressure transferred is the dilution factor. The canister is pressurized with UHP N2 or UHP He and is then allowed to equilibrate.

7.3.3. Working Gas Standard Prepared in Air Sample Bags - To prepare a working gas standard, an aliquot of the primary gas standard or of the pre-mixed primary gas standard is transferred to a clean air sample bag. The aliquot is transferred by direct injection with a volumetric gas-tight syringe. The air sample bag is filled with UHP N2 or UHP He to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

7.3.4. To calculate the concentration of the standard prepared:

$$\text{ppmv} = C_{ps} \times P_i / P_f$$

Where:

C_{ps} = concentration of compound in primary gas standard, ppmv

P_i = aliquot of primary gas standard used in psia or in mL (if using air sample bag)

P_f = final pressure of canister in psia or final volume in mL (if using air sample bag)

7.4. The preparation of each standard is recorded in the Laboratory Information Management System (LIMS).

7.5. Expiration date for standards and reagents are based on vendor specification. If no vendor expiration date is assigned, the laboratory assigns an expiration date of two years from the date of receipt. Refer to TestAmerica SOP WS-QA-0017 for further information on standards and expiration dates. Expiration dates must be documented on the gas cylinders. Expiration of working standards is six months.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample container, preservation techniques, and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements:

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Passivated Canister	2000 mL	None	30 days	EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air
Passivated Canister	2000 mL	None	30 days	Advisory – Active Soil Gas Investigations FINAL, April 30, 2012 (DTSC and LARWQCB)
Air Sample Bag	500 mL	None	72 hours	N/A

8.2. Passivated canisters used for sample collection must be certified clean (see SOP WS-QA-0032). A 7-micron filter should be placed on the inlet of the canister to protect the valve from particulates.

8.3. Samples should be shipped at room temperature, in packaging suitable to prevent puncture and exposure to light.

8.4. If air sample bags are to be shipped by aircraft, they should be filled about 75% full to allow for expansion during shipment.

8.5. The pressure of a canister should be recorded before and after sample collection in the field to help detect canister leakage and document proper sampling.

8.6. Samples are stored at room temperature.

8.7. **Important Note:** The information in this section is the minimum laboratory requirements and is communicated to the client via the Sample Acceptance Policy delineated in the section of the QAM that discusses Handling of Samples.

9. QUALITY CONTROL

9.1. Batch

A batch is defined as a set of up to 20 client samples of the same matrix processed using the same procedures and the same lot(s) of reagents within the same time period. A batch must contain a Laboratory Control Sample (LCS) and a Method Blank, but they do not count towards the maximum 20 samples in a batch.

9.1.1. Rerun of the same client sample is counted as part of the 20 in a batch (i.e., a client sample analyzed twice in the same batch must be counted as two client samples).

9.1.2. Field quality control (QC) samples (e.g., trip blanks, equipment blanks, and field duplicates) count as client samples; therefore, they add to the batch count.

9.1.3. Laboratory QC samples, including duplicates and clean canister blanks (screen cans), do not add to the batch count.

9.1.4. In some cases, an LCS Duplicate may be required by a client or program to provide batch precision. In that instance, the acceptance criteria and corrective actions appropriate for the LCS are applied.

9.1.5. The batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

9.1.6. Refer to the laboratory's QC Program document (WS-PQA-003) for further details of the batch definition.

9.2. Laboratory Control Sample

For each batch of samples, an LCS must be analyzed after the calibration standard and before the method blank and samples. The LCS is spiked with all the target analytes at concentrations within the calibration range of the method.

9.2.1. If any analyte is outside established recovery and precision control limits, the system is out of control and corrective action must occur. Corrective action typically includes reanalysis of the LCS or the batch. See also the troubleshooting guidelines in Section 11.5.

9.2.1.1. Repeated failures are an indication of a <u>systematic deficiency</u> that must be corrected.

9.2.1.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the LCS and/or LCSD after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to poor standard preparation, low standard pressure, closed standard valve, incorrect or broken sample line used, detector turned off, or low GC supply gas pressure.

9.2.1.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, the entire batch must be re-analyzed starting with a new initial calibration (ICAL) or a new continuing calibration verification (CCV), depending on whether maintenance or repair was performed.

9.2.2. The LCS/LCSD that failed acceptance criteria should be further evaluated as follows as per the QC program document (QA-PQA-003).

9.2.3. If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable reasons for not reanalyzing include evaluation of sporadic marginal exceedances (ME), or an elevated recovery (indicating a high bias) with samples non-detect for the failing analyte. Refer to the QC program document (WS-PQA-003) for more details regarding evaluation and acceptance of out of control LCS data.

9.2.4. Current LCS/LCSD control limits are stored in the LIMS. Control limits are subject to change based on periodic evaluation of LCS/LCSD control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.3. Method Blank

For each batch, an acceptable method blank must be analyzed. The method blank is analyzed after the calibration standards and LCS and prior to client samples. The method blank is typically UHP He collected in either a passivated canister or an air sample bag, or may be obtained directly from the laboratory's He supply line. The method blank for H2 and He analysis is UHP N2. The method blank for CH4 (FID), C2H4, C2H6, and C2H2 analysis is UHP He.

9.3.1. The Method Blank must <u>not</u> contain any analyte of interest ≥ RL (or ≥1/2 RL, as dictated by the QSM or project-specific requirements. Otherwise, the Method Blank is further evaluated and corrective actions must be performed,

as stated below. See troubleshooting guidelines in Section 11.5.

9.3.1.1. Repeated failures are an indication of a systematic deficiency that must be corrected.

9.3.1.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the method blank after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to sub-ambient method blank pressure, closed method blank container valve, incorrect or broken sample line used, detector turned off, or low GC supply gas pressure.

9.3.1.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, the entire batch must be re-analyzed starting with a new ICAL or a new CCV, depending on whether maintenance or repair was performed.

9.3.2. Re-analyze the Method Blank once to determine if an error or an anomaly occurred during sample analysis. If the re-analysis is acceptable, then the Method Blank can be considered in control.

9.3.3. If there are no results greater than the RL in the samples or if the results in the samples are greater than 10X the Method Blank level, the data may be reported with qualifiers. In this case, the elevated Method Blank result is not believed to impact data quality. The anomaly must be reported in an NCM.

9.3.4. If there are results greater than the RL in the samples and if these results are less than 10X the Method Blank level, the samples must be re-analyzed.

9.3.4.1. If re-analysis is not possible due to limited sample volume or other constraints, the Method Blank is reported and all associated samples are flagged. The client must be consulted. The anomaly must be reported in an NCM. The laboratory Project Manager (PM) must record the client's decision in the NCM.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Initial Calibration

10.2.1. Instruments must be calibrated at initial setup and as needed thereafter, and at least annually. More frequent calibrations must be performed if project-specific requirements dictate.

10.2.2. Prior to sample analysis, an ICAL curve consisting of a minimum of five points at different concentrations is analyzed to determine the linear working range of the analytical system for each target analyte. The lowest calibration level for each target analyte must be at or below the RL. The highest calibration level is considered to represent the upper range of the analytical system for the corresponding target analyte.

10.2.3. The acceptability of the calibration curve for each target analyte is determined by evaluating the percent relative standard deviation (%RSD) of the average CF or RF calculated from the different calibration levels, or by evaluating the correlation coefficient (r) or the coefficient of determination (r2) of the curve.

10.2.3.1. For calibration by average RF, the calibration is deemed acceptable for a given analyte if the %RSD is ≤ 25 (or ≤ 20 for EPA 3C analysis). Peak area is used for this determination.

10.2.3.2. A calibration curve based on linear regression fit is acceptable if r ≥ 0.995 and a minimum of six points is required.

10.2.3.3. A calibration curve based on quadratic (second-order) fit is acceptable if r2 ≥ 0.990 and a minimum of six points is required.

Note: The data acquisition system used for this GC analysis requires forcing the analytical curve through zero. Otherwise, all detections below the lowest point of the calibration curve will not be quantitated.

10.2.4. If the %RSD, r, or r2 does not meet the required acceptance criteria, the corresponding curve is considered invalid and corrective action must be performed. See troubleshooting guidelines and techniques in Section 11.5.

10.2.4.1. Repeated failures are an indication of a systematic deficiency that must be corrected.

10.2.4.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the specific calibration level(s) after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to poor standard preparation, low standard pressure, closed standard container valve, incorrect or broken sample line used, detector turned off, or low GC supply gas

pressure.

10.2.4.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, all calibration levels must be re-analyzed and re-evaluated to determine if the %RSD, r, or r2 meets acceptance criteria.

10.2.5. A new ICAL is required when situations like, but not limited to, the following are encountered:

- After changes are made to the original ICAL instrument configuration
- After replacement of analytical columns
- After replacement of detectors

10.2.6. The analyst may elect to drop points from the calibration curve to improve subsequent quantitation, in accordance with Policy CA-T-P-002, Selection of Calibration Points.

10.2.7. Initial Calibration Verification (ICV)

Each new multi-point calibration must be verified using a second-source standard. The second-source standard, as quantitated against the new calibration curve, must have recoveries between 80% and 120% for each target analyte. If these criteria are not met, the following corrective actions must be performed:

- Rerun the second-source check standard.
- Re-prepare or acquire a new standard.
- Evaluate instrument conditions or perform maintenance or repair, if needed.
- Re-analyze a new ICAL.

10.2.8. An acceptable ICAL and ICV is documented using a GC Initial Calibration Curve Review Checklist (see Attachment 6). This checklist is submitted to the Department Manager, or designee, for second-level review and signature. Only after successful completion of first and second-level reviews may the analysis be reported from the new ICAL.

10.3. Continuing Calibration Verification

10.3.1. A batch may be started after an approved ICAL and ICV have been completed.

10.3.2. An opening CCV standard is analyzed at the onset of each batch, or every 24-hour shift, whichever comes first, to verify the linearity of the ICAL. The CCV will contain each analyte of interest, on each applicable detector, a different source from that used in the ICV. The system is considered in control if the percent difference (%D) between the RF of the analyte in the CCV and the average RF of the analyte in the ICAL is ± 20 (for both ASTM D1946 and EPA 3C analysis).

10.3.3. If the CCV fails acceptance criteria, corrective actions must be performed. Per the NELAC Standard (Quality Systems, June 5, 2003, Section 5.5.5.10e, pages 216 and 217 of 324) and TNI Standard (EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2e, page 92), if routine corrective action procedures fail to produce a second consecutive (immediate) CCV within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive CCVs, or a new ICAL must be generated. See troubleshooting guidelines in Section 11.5.

10.3.3.1. Repeated failures are an indication of a systematic deficiency that must be corrected.

10.3.3.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the CCV after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to poor standard preparation, low standard pressure, closed standard valve, incorrect or broken sample line used, detector turned off, or low GC supply gas pressure.

10.3.3.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, the CCV (if this still meets the NELAC or TNI requirement stated in Section 9.2.4.3) may be re-analyzed. Otherwise, a new ICAL is required.

10.3.4. Following the last injected sample, the batch is closed with a passing CCV, which must also meet the same acceptance criteria as the opening CCV. Per the NELAC Standard (Quality Systems, June 5, 2003, Section 5.5.5.10c, page 216 of 324) and TNI Standard (EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2c, page 92), acceptable opening and closing CCVs must bracket reportable samples.

10.3.4.1. If the closing CCV fails acceptance criteria, follow the corrective action procedures defined in Section 10.3.3.

10.3.4.2. If any target analyte or target group in the closing CCV exceeds acceptance criteria and the target analyte or target group was ND in the associated samples, no further corrective action is required. However, the nonconformance must be reported in an NCM.

10.3.4.3. If any sample result is reported from a batch where the closing CCV failed acceptance criteria, the CCV failure must be documented in an NCM. The NCM must indicate the reason why the sample results, in the best judgment of the analyst, are being reported under a failed closing CCV.

10.3.5. If sample analysis must be halted for more than an 8-hour hour period, an opening CCV must be analyzed prior to continuance of the batch, to ensure that instrument conditions have remained stable.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Preparation

11.2.1. For air sample bag samples, the air sample bag is checked for damage and is analyzed as received.

11.2.2. For passivated canister samples, the initial pressure is checked by attaching the process meter line connector to the passivated canister. The process meter line connector must be rinsed before use, with the pressurization gas (UHP N_2 or UHP He, if requested) by physically holding it against the gas outlet and flushing for 10 seconds, as this avoids possible carry-over concerns from high concentration samples. With the process meter line connector attached, the passivated canister valve is opened briefly and the pressure is recorded. If the pressure is less than 6 psig, the passivated canister is pressurized to 10 psig with the pressurization gas. The initial and final pressure must be recorded in

the Canister Pressurization Logbook (see Attachment 7) and in the individual Canister Field Data Record (see example form in SOP WS-QA-0032).

11.2.2.1. If there is an associated analytical test (e.g., EPA TO-14A or EPA TO-15) to the sample that requires use of a mass flow controller to measure injected sample volume, the passivated canisters should be pressurized with UHP N2 only.

11.2.2.2. If UHP N2 is used, then the final GC results must be corrected for N2 content (see Section 11.7).

11.2.2.3. If N2 is also a target analyte, a back calculation/correction factor is used (see Section 11.7 and 12.8.9).

11.2.3. When the passivated canister vacuum/pressure is increased, a dilution factor is calculated and is applied to the results. The calculation is provided in Section 12.8.6.

11.2.4. Pressurizing canister sample with UHP He is an option, if requested by clients. Pressurizing samples with UHP He will eliminate the error associated with N2 correction. EPA TO-14A or EPA TO-15 analysis on samples pressurized with UHP He will require a flow correction factor.

11.3. Calibration

11.3.1. Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

11.3.2. Refer to Section 10 for details regarding instrument calibration.

11.4. Sample Analysis

The calibration standards and the sample QC are analyzed in the same manner as client samples. After the calibration standards are analyzed and evaluated (Section 10), the sample QC is analyzed and evaluated (Section 9), all prior to client sample analysis.

11.4.1. Recommend instrument conditions are presented in Attachment 2. If, after instrument maintenance or repair, these parameters change, then the updated parameters must be listed in the instrument maintenance log. The appropriate QC is performed prior to analysis of samples to verify method performance.

11.4.2. For analysis using EPA 3C, all samples must be analyzed in duplicate. The results are acceptable when the peak areas for two consecutive injections

agree within 5% of their average (<10% difference between the sample runs). Note that only client samples are analyzed in duplicate. All calibration and sample QC are analyzed once.

11.4.2.1. If the duplicate runs exceed the acceptance criteria, run the sample one more time to confirm that the excessive variation is due to the sample matrix/container and not the analytical system. Otherwise, perform corrective action based on the investigated cause of the variation. See similar corrective actions defined in Section 9.

11.5. Troubleshooting Guidelines and Techniques

11.5.1. Low pressure in canisters containing the standards or problems with carrier/detector gas supply: Always confirm that adequate pressure remains in the canisters and that the instrument gas supplies are sufficient before working on the instrument hardware.

11.5.2. Low Response: Causes are typically leaking detector.

11.5.3. Baseline Noise: Check for supply gas contamination and leaking fittings. Carrier gas filters may need to be changed, including the pencil filters inside the GC. Sample carry-over or contamination may also be an issue and baking the system while flushing sample lines will remove most carry-over.

11.5.4. Valve Switching Issues: Excessive baseline disruptions can be caused by valve actuation when the columns are not properly balanced. Balance the columns by adjusting the carrier restrictors so that head pressure does not change when the column is taken offline.

11.5.5. Instrument Issues: If data loss or error messages are encountered, consult the instrument troubleshooting guidance found in the operator's manual. The manual is in the help section of the GC software.

11.6. Maintenance or Repair of Analytical Instruments or Support Equipment

11.6.1. When analytical instruments or support equipment require repair or maintenance, they shall be taken out of operation or otherwise isolated, and tagged as 'out-of-service' until such a time as the repairs or maintenance have been made and the instrument or support equipment can be demonstrated as operational by calibration and/or verification or other tests to demonstrate acceptable performance. Details on the tag-out procedures to be followed may be found in the section of the QAM that discusses Equipment and Calibrations.

11.6.2. Schedule for routine maintenance of analytical instruments may be found in

Attachment 4.

11.6.3. All maintenance or repair must be documented in the Instrument Maintenance Logbook (see example in Attachment 8).

11.7. Monochrom

11.7.1. All samples in passivated canisters that have been pressurized with UHP N2 and require N2 as a compound of interest need to be ran through the Monochrom program. Monochrom is used to recalculate N2 by subtracting out the N2 used to pressurize the samples. Monochrom then generates graphics and uploads the graphics into TALs and updates the new corrected N2 concentration. All samples that require D3588/EPA 3C also need to be run through Monochrom for specific calculations needed for those methods. Refer to the Monochrom manual located on the intranet for specific instructions on how to use the Monochrom program.

12. CALCULATIONS/DATA REDUCTION

12.1. After the analytical run has ended, a real-time review of the data is performed to determine if a successful analysis has been achieved. Peaks which exceed the upper calibration range and/or which exceed the signal output of the detector must be diluted and re-analyzed.

12.2. The chromatogram/data is reviewed to determine if the proper dilution has been performed. If the sample was analyzed at the proper dilution, the chromatogram/data is evaluated to determine if proper peak identification and integration was performed.

12.3. A target compound is identified by comparison of the sample analyte RT with that of the standards analyzed within the corresponding batch.

12.4. When a compound has been identified, that compound will be quantitated based on its RF from the ICAL, the integrated area of the peak as determined by the instrument data system, the dilution factor, and the N_2 correction (if applicable).

12.4.1. The data system automatically quantitates the sample results. If a canister sample was pressurized before analysis, the results must be multiplied by the DF (see Section 12.8.7). Correction for N2 (if applicable) must also be performed (see Section 12.8.9).

12.4.2. If, in the best judgment of the analyst, the instrument data system integration is incorrect, then the analyst may manually integrate the peak. See Section 12.7.

12.5. The laboratory reviews the total or summation of the fixed gases reported as part of the data validation process. The review is a secondary check to confirm that valid data acquisition occurred during analysis. The assessment of 95-100% v/v criteria can provide useful information when the analysis is for the full list of fixed gases, but is not required when a subset of compounds is analyzed or reported. If the total fixed gases result is below this range, it is possible that the difference is due to the amount of non-analyzed components in the sample. This case must be investigated. Screening data, NMOC data, subsequent H_2 and He analysis, sulfur compounds analysis, and/or other information should be considered. If the sample containers were pressurized with UHP N_2, then they should be evaluated for error associated with UHP N_2 pressurization. Samples that do not meet the required total fixed gases acceptance criteria and which deviation is not yet accounted for, must be re-analyzed at least once.

12.5.1. An investigation of the deviation from the total or summation acceptance criteria may result into transducer recalibration, instrument maintenance, and/or instrument recalibration. Samples must be re-analyzed after the condition has been remedied. The area supervisor, or designee, should be notified as soon as possible when re-analysis is expected to be outside the sample holding time.

12.5.2. Every time the acceptance criteria for the total or summation of fixed gases result are not met, the deviation and the resulting investigation must be reported in an NCM. The same reporting procedure must be followed for unexplained deviation.

12.6. Second-column confirmation for the results is not required due to the unique separation properties of the multiple column analytical system and the limited possibilities of non-analyte interference.

12.7. All manual or re-integration of chromatograms must be documented in accordance with TestAmerica Corporate SOP CA-Q-S-002. Documentation includes, at a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration, dated, and initialed. All manual integrations must undergo a secondary-level review.

12.8. Calculations

12.8.1. Calculation for RF

$$\mathrm{RF} = \frac{\text{Peak Area in Standard}}{\text{Concentration of Standard}}$$

12.8.2. Calculation for RPD

$$\text{RPD} = \frac{|\text{Value A - Value B}|}{\text{Average of Values}} \times 100$$

12.8.3. Calculation for %RSD

$$\%\text{RSD} = \frac{\text{Std. Dev of RFs}}{\text{Mean of RFs}} \times 100$$

12.8.4. Calculation for %D

$$\%D = \frac{RF\ ICAL - RF\ CCV}{RF\ ICAL} X 100$$

12.8.5. Calculation for %Rec

$$\%\,\text{Rec} = \frac{Amount\ cpd.\ recovered}{Amount\ cpd.\ spiked} X\ 100$$

12.8.6. Calculation for Pressure DF

$$DF = \frac{Y_a}{X_a}$$

Where:

X_a = absolute canister pressure before dilution (initial pressure)
Y_a = absolute canister pressure after dilution (final pressure)

12.8.7. Calculation for determining concentration of compound in sample, using average RF

$$\text{Conc. Compound} = \frac{\text{Area Compound}}{\text{Ave RF for Compound}} \times \text{DF}$$

12.8.8. Reporting units are typically % v/v for ASTM D-1946 and EPA 3C analysis. If results are to be reported in units of ug/L (also mg/m³), use the following equation:

$$\text{Result (ppmv)} \times \frac{\text{Molecular weight of compound}}{24.45} = \text{ug/L}$$

Note: 24.45 is the volume of ideal gas at 25º C and 1 atmosphere
1% v/v = 10000 ppmv

12.8.9. Calculation for N_2 results in samples pressurized with N_2:
Total amount of N_2:

$$T_{N2} \text{ (as analyzed)} = S_{N2}(I/F) + 100((F-I)/F)$$

Where:

T_{N2} is the result % v/v of N_2 as analyzed (no DF applied)
S_{N2} is the N_2 in the sample (no DF applied)

I and F are the initial pressure and the final pressure, respectively, of the sample during pressurization (psia)

100 is the percent concentration of N_2 added

Rearranging,

$$S_{N2} = (T_{N2} - 100((F-I)/F))F/I$$

Or, in final form with the DF applied:

N_2 reported, % v/v = N_2 total (as analyzed, with DF) – [100 x (DF-1)]

12.9. No conversion of the analytical results to standard conditions is made.

12.10. Calculations for D1945 (Natural Gas)

This calculation is performed automatically by Monochrom.

Before performing the calculations for D1945, the following parameters must be determined:

D1946	TO3	EPA 15_16
CO2, O2, N2, CH4, CO, H2, He	Ethane, Propane, Butane, Pentane, C5 Range, C6 Range, C7 Range, C8 Range, C9 Range, C10+ Range	H2S (optional)

12.10.1. Results for each of the above parameters are normalized by first summing the raw % v/v results for each parameter to determine the total %. The values are then normalized:

$$\text{Normalized Result} = \frac{\text{Raw Result} \times 100}{\text{Total \%}}$$

When the normalized results are summed, they should equal 100%.

Non-detect values are presumed to be 0 for the purposes of this calculation.

12.11. Calculations for D3588

This calculation is performed automatically by Monochrom.

Before performing the calculations for D3588, the following parameters must be determined, and the normalization as described above performed.

D1946	TO3	EPA 15_16
CO2, O2, N2, CH4, CO, H2,	Ethane, Propane, Butane,	H2S (optional)

He	Pentane, C5 Range, C6 Range, C7 Range, C8 Range, C9 Range, C10+ Range	

12.11.1. Three properties are determined for D3588 – Ideal Gross Heating Value, Ideal Net Heating Value, and Specific Gravity. For each property, the contribution from the measured values is calculated by using the property constant and the normalized result:

$$\text{Contribution} = \frac{\text{Normalized Result} * \text{Property Constant}}{100}$$

Units for Gross Heating Value and Net Heating Value are btu/cubic ft.

Parameter	Gross Heating Value Constant	Net Heating Value Constant	Specific Gravity
Carbon Dioxide	0	0	1.5196
Oxygen	0	0	1.1048
Nitrogen	0	0	0.96723
Methane	1010	909.4	0.55392
Carbon Monoxide	320.5	320.5	0.968
Ethane	1769.7	1618.7	1.0382
Propane	2516.1	2314.9	1.5226
n-Butane	3262.3	3010.8	2.0068
n-Pentane	4008.9	3703.9	2.4912
C5 Balance	4008.9	3703.9	2.4912
C6 CPDS	4755.9	4403.9	2.9755
C7 CPDS	5502.5	5100.3	3.4598
C8 CPDS	6248.9	5796.2	3.9441
C9 CPDS	6996.5	6493.6	4.4284
>/= C10 CPDS	7742.9	7189.9	4.9127
Hydrogen Sulfide	637.1	586.8	1.1767
HYDROGEN	324.2	273.93	0.6960
Helium	0	0	0.13820

12.11.2. Once individual values are calculated, the results for each property are summed and reported.

12.10.12.12. Technical Data Review

Technical data review is performed in accordance with Policy WS-PQA-012, and is documented utilizing review checklists. Examples of appropriate review checklists are found in Attachments 5 and 6.

12.10.1.12.12.1. One aspect of technical review is to ensure that the test instructions are clear, and that all project-specific requirements have been understood and followed. If directions to the analyst are not clear, the analyst must consult the Department Manager or the appropriate PM, who must clarify the instructions.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for air, soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the CCV/LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Expired standards will be part of the Lab Pack waste stream. They will be identified as expired, stored under the manufacturer's recommended conditions, and then packed for disposal as outlined in SOP WS-EHS-0001.

15.1.1. Gas standards in non-returnable, non-refillable cylinders, such as Scotty® Transportables, are slowly vented in the fume hood. Once empty, they are turned over to the Hazardous Waste Specialist and damaged (e.g. a hole drilled into the cylinders so they cannot be reused, then they are disposed or recycled.

15.1.2. Gas standards in returnable, refillable cylinders are returned to the manufacturer.

15.2. Air sample bags are slashed in a hood and allowed to completely vent, then placed into an orange high VOA lab trash can. When the can is full or after no longer than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment..

16. REFERENCES/CROSS REFERENCES

16.1. ASTM D 1946-90 (Re-approved 2000), Standard Practice for the Analysis of Reformed Gas by Gas Chromatography.

16.2. EPA 3C, Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from stationary sources, 40 CFR Chapter 1, Part 60, Appendix A.

16.3. TestAmerica Sacramento QAM, current revision

16.4. TestAmerica Corporate Environmental Health and Safety Manual CW-E-M-001, current revision

16.5. Advisory – Active Soil Gas Investigations, January 28, 2003 (DTSC and LARWQCB)

16.6. EPA/600/R-04/003, 2003 NELAC Standard, June 5, 2003

16.7. The NELAC Institute (TNI) Standard 2009, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis

16.8. TestAmerica Corporate SOP CA-Q-S-005, Calibration Curves (General), current revision

16.9. TestAmerica Corporate SOP CA-Q-S-002, Manual Integrations, current revision

16.10. TestAmerica Corporate SOP CA-Q-S-006, Detection Limits, current revision

16.11. TestAmerica Sacramento Safety SOP WS-EHS-0001, Sample and Chemical Waste Characterization, Collection, Storage and Disposal, current revision.

16.1.16.12. ASTM D 1945-03 (Re-approved 2010), Standard Test Method for Analysis of Natural Gas by Gas Chromatography

16.13. ASTM D 3588-98 (Re-approved 2011), Standard Practice for Calculating Heat Value, Compressibility Factor, and Relative Density of Gaseous Fuels.

17. METHOD MODIFICATIONS

17.1. ASTM D-1946

17.1.1. The method uses a calibration method in which a separate standard is run closely approximating each sample's concentration for target analytes. TestAmerica Sacramento uses the more widely accepted method of initial multi-point and continuing calibrations to define linear response.

17.1.2. TestAmerica Sacramento does not normalize sample results to 100%, unless required by the client. Normalizing assumes a zero contribution of all other components not analyzed (e.g., H2, He, hydrogen sulfide, etc.), and can affect results significantly.

17.1.3. Commercially-prepared standards that have the industry standard analytical uncertainty of 2-5% are used.

17.1.4. Standards may be prepared in N_2 as well as in He.

17.1.5. The method specifies use of a TCD.

17.1.6. Due to the multi-detector, multi-valve configuration of TestAmerica Sacramento's analytical instrument for fixed gases analysis, one or more sample loops, not necessarily equal to 0.5 mL, may be used.

17.2. EPA 3C

17.2.1. The method specifies use of at least three concentration levels for the multi-point calibration, spanning the range of suspected sample concentrations. TestAmerica Sacramento uses a minimum of five-point calibration on each applicable detector for all standard analyte components, to define the reporting range.

17.2.2. TestAmerica Sacramento does not use correction for moisture content. This would require additional client measurements to be supplied, and at most would result in a 2-3% correction in reported values.

17.2.3. TestAmerica Sacramento does not use correction for temperature at sampling. As the laboratory supplies fully evacuated sample containers for sampling, any field temperature does not affect results. Laboratory temperature is constant for sample pressurization and analysis.

17.2.4. The method specifies that "helium must be used to prepare calibration gases" so that detector response is consistent. TestAmerica Sacramento also uses UHP N2, UHP helium, or zero-grade air to prepare calibration gases. Detector response is unaffected by the diluent gas since the analytical system separates the diluent gas from the standard components prior to detection. Furthermore, sample injection size is determined by a sample loop and not a mass flow controller.

17.2.5. The method specifies that if two consecutive sample injections do not agree within 5% of their average, then run additional samples until consistent area data are obtained. TestAmerica Sacramento requires that only at least one additional injection be analyzed. Refer to Section 11.4.5.

18. ATTACHMENTS

18.1. Attachment 1: Target Analytes and Reporting Limits

18.2. Attachment 2: GC Conditions – Agilent 7890A

18.3. Attachment 3: Nominal ICAL Concentrations

18.4. Attachment 4: Schedule for Routine Maintenance of Analytical Instrument

18.5. Attachment 5: Example GC Initial Calibration Curve Review Checklist

18.6. Attachment 6: Example GC Technical Data Review Checklist

18.7. Attachment 7: Canister Pressurization Logbook (Example Page)

19. REVISION HISTORY

19.1. WS-GCA-0020, Revision 1.2, Effective 07/25/2014

19.1.1. Added references to D3588 and D1945 in the title and Section 16.

19.1.2. Sections 12.10 and 12.11 were inserted to support the calculations for D3588 and D1945.

19.1.3. Editorial Changes.

19.2. WS-GCA-0020 Revision 1.1, Effective 05/28/2014

19.2.1. Changed Section 2.1 to include – "Carbon monoxide (CO) is last to elute from the HaySep, bypassing the isolated Mol Sieve, then goes to the TCD. Ultra high purity (UHP) helium (He) is used as the carrier gas.

19.2.2. Updated Table in Section 5.2 to include Carbon Monoxide.

19.2.3. Editorial changes.

19.3. WS-GCA-0020, Revision 1, Effective 6/19/2013

19.3.1. Included reference to Chrom as the data processing software in Section 6.1

19.3.2. Change references to SOP LA-QAS-002 and LA-SRA-002 to refer to the appropriate Sacramento SOPs (WS-QA-0017 and WS-QA-0032, respectively).

19.3.3. The first sentence of Section 12.5 has been revised from "The laboratory acceptance criteria for the total or summation of the fixed gases reported must be equal to or greater than 95% v/v, not to exceed 105% v/v." to "The laboratory reviews the total or summation of the fixed gases reported as part of the data validation process. The review is a secondary check to confirm that valid data acquisition occurred during analysis. The assessment of 95-100% v/v criteria can provide useful information when the analysis is for the full list of fixed gases, but is not required when a subset of compounds is analyzed or reported."

19.3.4. Inserted "required" into the last sentence of Section 12.5.

19.3.5. Replaced Attachments 5, 6, and 7 with Sacramento-specific examples.

19.3.6. Removed Attachment 8 (example maintenance logbook).

19.3.7. Updated Attachment 3 with current calibration concentrations.

19.3.8. Inserted section 9.1.4, "In some cases, an LCS Duplicate may be required by a client or program to provide batch precision. In that instance, the acceptance criteria and corrective actions appropriate for the LCS are applied."

19.4. WS-GCA-0020, Revision 0, Effective 04/01/2013

19.4.1. This is the first version of this SOP.

Attachment 1: Target Analytes and Reporting Limits

Compound	RL, % v/v[1]
Carbon Dioxide	0.50
Carbon Monoxide	0.010
Methane	0.50
Nitrogen	1.0
Oxygen	0.20
Helium	0.10
Hydrogen	0.010

Attachment 2: GC Conditions – Agilent 7890A

Fixed Column Oven: 100°C
FID: Oven 250°C, FID Range = 12, Time Constant = Slow
TCD: Oven 130°C, Filament 210°C, Range = 0.5, Polarity = positive, Time constant = slow
Reduction Catalyst: 380°C
Sample Lines and A/S valve at 80°C
Valve Ovens at 120°C
Fixed FF = 20 mL/min, BF = 30 mL/min w/ He reference at 20 mL/min
H_2/He FF=20 mL/min, BF = 20 mL/min w/ N_2 reference at 20 mL/min
FID H_2 20 mL/min + 10 mL/min Red Cat H_2
FID Air = 300 mL/min
Initial time t = 0, all events off
0.01 min: Sample flow starts
1.7 min Sample flow stops with 0.2 min before loop vent
2 min Sample loop injection
3.05 min Mol Sieve Column Isolation
3.5 min H_2/He stripper BF for 4 min relay event time
6.5 min Fixed BF
6.7 min Fixed Mol Sieve in line
Additional events include catalyst recharge and sample advance pre-purge

[1] Note that RLs are subject to change based on annual MDL studies.

Attachment 3: Recommended Nominal ICAL Concentrations (%v/v)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Carbon Dioxide	0.01	0.02	0.05	0.2	1.0	5	10	50	100
Carbon Monoxide	0.01	0.02	0.05	0.2	1.0	5	10	50	100
Methane - TCD	0.50	1.0	5.0	10	50	-	-	-	-
Nitrogen	1.0	10	30	80	100	-	-	-	-
Oxygen	0.2	1.0	10	20	30		-	-	-
Helium	0.02	0.1	0.2	1.0	10	50	-	-	-
Hydrogen	0.010	0.1	0.5	1.0	10	50	-	-	-

Attachment 4: Schedule for Routine Maintenance of Analytical Instrument

Frequency	Maintenance Item
Daily	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level with analysis of blanks. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.
Quarterly	Oxidation and Reduction Catalysts: Perform leak checks. Replace/condition when poor response is observed.
As needed	All Detectors: Clean when baseline indicates contamination or when response is low. Perform periodic leak checks (quarterly). Replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds). Bake trap to correct for high background. Replace gas supply. Clean moisture filters. Bake column.

Attachment 5: Example GC Initial Calibration Curve Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

GCA ICAL Review Checklist

Instrument ID: _____ Method: _____

Work List ID: _____ Analysis Batch(es): _____

Analytes Included in this ICAL: _____

ICAL Review	1st Level	2nd Level	N/A
1. Method locked in Chrom.			
2. ICAL is set as most recent method in Chrom.			
3. RTC Marker included for and RT TO3 windows set correctly.			
4. Standards used in ICAL are current.			
5. No analytes have saturated peaks.			
6. All peaks correctly identified and within retention time windows.			
7. Manual Integrations reviewed and appropriate.			
8. All analytes meet %RSD or linear regression requirements.			
9. Any levels dropped are reviewed and appropriate.			
10. ICV meets requirements and is run after the ICAL.			
11. RFs are calculated correctly. Perform Manual calculation.			
12. ICAL meets requirements stated in SOP.			
13. Chromatograms are uploaded/reviewed in TALs.			
14. ICAL is locked in TALs.			

Curve Valid for:	Yes	No	Criteria
Standard SOP			RSD ≤20%, r≥ 0.995 (intercept <1/2RL for 8081,8082, NQ) and (<RL for 8330) and r>0.990 (intercept <RL for 8015), ICV≤15%, PEM ≤15%
DOD QSM V3,V4, V5			RSD ≤20%, r≥ 0.995, ICV≤20%, PEM ≤15%

1st Level Reviewer: _____ Date: _____

2nd Level Reviewer: _____ Date: _____

Comments: _____

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Attachment 6: Example GC Technical Data Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

Air GC Data Review Checklist

Job Number(s): _____ Instrument/Work List ID(s): _____

TALS Analytical Batch(es): _____ ICAL Batch(es): _____

☐ TO3 ☐ TO12 ☐ 1946/3C ☐ EPA 15/16

Review Items	--- Level 1 --- Yes	No	N/A	Level 2
Initial Calibration				
1. Is ICAL locked in Chrom and in TALS?				
2. If RRF used for ICAL, did it meet acceptance criteria? ☐ 20% (8000 series) ☐ ≥ 0.990 (EPA 1516) ☐ 30% (TO-12)				
3. Is ICV properly linked in TALS for Tier 4 jobs ?				
Continuing Calibration				
1. Did %Drift (%D) meet method criteria (30% TO-12) (15% EPA 15/196) (20% QSM TO-3/1946)?				
2. Are compounds within retention time windows?				
3. Does CCV frequency meet criteria (10 samples QSM) or every 20 samples (standard work)?				
4. RTC present for TO3 analytes?				
Client Samples & QC Sample Results				
1. Was the correct analysis performed & were project instructions followed?				
2. Were preparation and analysis completed within holding times?				
3. Are preparation weights/volumes entered correctly?				
4. Have Chromatograms been uploaded/reviewed?				
5. Are positive results within calibration range?				
6. Are any dilutions due to matrix? (Requires NCM if "yes.")				
7. Are all positive results within RT windows, both columns if 2 required?				
8. Are target constituents in LCS/LCSD within control limits?				
9. All target analytes in MB < RL (< ½ RL for DoD)? (Requires NCM if "no.")				
10. Do results (e.g. dilutions) make sense?				
11. Are all manual integrations appropriate and documented properly?				
12. Are nonconformances documented as NCMs?				
13. Are all QC samples properly linked in TALS?				
14. Have all flags been reviewed for appropriateness?				
15. Are all Chrom graphics and reports reviewed/uploaded in the analytical batch?				
16. Has QC checker been run?				
17. NCM numbers:				

1st Level Reviewer: _____ Date: _____

2nd Level Reviewer: _____ Date: _____

Comments: _____

_____

_____

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Attachment 7: Canister Pressurization Logbook (Example Page)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

Canister Pressurization Log

Date	Sample ID	Can #	Initial Press. (psig)	Initial Press. (psia)	Final Press. (psig)	Final Press. (psia)	Gauge ID	Initial	Comments
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		

A = Gauge CD5437; B = Gauge _____ [Calculation: psig + 14.7 = psia]

QA-813 r1 GEC 7/21/2014 PAGE 1 Reviewed by /Date: _____

Title: Acid Digestion Of Aqueous Samples by SW846 [Methods 3005A, 3010A, 200.7-4.4, 200.8-5.4]

Approvals (Signature/Date):

12/3/14	12/5/14
Thep Phomsopha — Date Inorganic Supervisor	Joe Schairer — Date Health & Safety Manager / Coordinator
12/4/2014	12/4/14
Lisa Stafford — Date Quality Assurance Manager	Robert Hrabak — Date Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP – method 6010B, 6010C, 200.7-4.4), and Inductively Coupled Plasma Mass Spectrometry (ICPMS – method 6020,6020A) using SW846 3005A/3010A, 200.8-5.4, and 200.7-4.4 sample preparation protocols.

1.2. This SOP provides procedures applicable to the preparation of total recoverable, dissolved, and total metals in ground water, surface water, saline water, and wastewater. It also applies to samples prepared by TCLP extraction.

1.3. Tables I and II show a list of the analytes that are analyzed at the Sacramento facility and that are evaluated relative to method performance per NELAC criteria.

2. SUMMARY OF METHOD

2.1. Method 3005A is applicable to the preparation of total recoverable metals and dissolved metals by ICP and ICPMS. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to the original volume.

2.2. Method 3010A is applicable to the preparation of total metals for ICP analysis. A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary) and brought up to the original volume.

2.3. Method 200.7-4.4 and Method 200.8-5.4 are applicable to the preparation of total recoverable metals by ICP and ICPMS. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to the original volume.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Dissolved Metals: Metals present in a sample after passing through a 0.45 um membrane filter. The sample should not be preserved prior to filtration. Once filtered the sample must be preserved to pH <2 with the addition of concentrated nitric acid.

3.4. Total Metals: metals present in an unfiltered, preserved sample followed by more aggressive digestion with nitric and hydrochloric acids (method 3010A).

3.5. Total Recoverable Metals: metals present in an unfiltered or dissolved sample following treatment with hot, dilute acid (method 3005A).

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing lab ware, wearing improper gloves, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix A for additional contamination control guidelines.

4.3. Boron and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate

safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Vinyl gloves provide adequate protection against the chemicals and reagents typically used during this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.1.2. The use of vacuum systems during filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.3. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.4. The acidification of samples containing reactive materials may result in the release of toxic gasses, such as cyanides or sulfides. Acidification of samples must be done in a fume hood.

5.1.5. When digesting samples on a hot plate or digestion block, heat protective gloves and/or hot tongs must be used when handling containers. Ensure that digestion containers are arranged safely and are not overcrowded.

5.1.6. Filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic break or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustment when there are numerous samples to be processed.

5.1.7. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.1.8. Acid washing of glassware is classified as a high-risk activity. A face shield must be worn over safety glasses or safety goggles during this process.

5.1.9. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and

reagents are being handled.

5.1.10. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, prepared, digested and cooled in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.11. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Digestion block capable of maintaining a temperature at approximately 95°C.

6.2. Calibrated thermometers that cover a temperature range of 0-200°C.

6.3. Disposable graduated block digestion tubes.

6.4. Whatman No. 41 filtration paper.

6.5. Bottle-top dispensers.

6.6. Adjustable air displacement pipettes.

6.7. pH indicator strips (pH range 0 - 14).

6.8. Polyethylene storage bottles.

6.9. Vacuum flask with filtration assembly.

6.10. Vacuum pump.

6.11. 0.45 um membrane filters.

7. REAGENTS AND STANDARDS

7.1. Use reagent grade chemicals in all tests. "Certificates of Analysis" should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.2. Reagent water is produced by a Millipore nanopure system (SOP WS-QA-0014). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as certified custom solutions (Tables I, II). All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. All stock standards must be labeled with the date of receipt and the date the standard is opened.

7.4. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the

custom solution, the lab must purchase a solution from a designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Nitric acid, concentrated: Analytical reagent grade

7.6. Hydrochloric acid, concentrated: Analytical reagent grade

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Samples submitted for "total recoverable" or "total" metals analysis should be preserved in the field to a pH <2 with the addition of HNO_3 after sampling. Prior to sample preparation every sample must be tested for proper preservation using pH paper.

8.3. For dissolved metals analysis, the samples should be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field or within 24 hours of collection. For filtration done by the laboratory the sample must be preserved with HNO_3 immediately following filtration.

8.4. Filtration and preservation done by the lab analyst must be documented by a NCM in the prep batch.

Note: If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be prepared within the same time frame. A method blank (MB) and a laboratory control sample (LCS) or duplicate control sample (LCS/LCSD) must be prepared as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One method blank (MB) must be prepared for every 20 samples. A method blank consists of reagent water processed through all of the steps, and at the same time as the associated samples. If a method blank exceeds +/- the reporting limit for a given analyte than the samples associated with that batch must be re-prepared. The exception

is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo. For samples requiring laboratory filtration and preservation, the method blank must also be filtered and preserved. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples. A LCS consists of reagent water spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared except when a LCS exhibits high recovery. For such a case, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3.1. For methods 6010B and 6020A a Low Level Control Sample (LLCS) must be prepared for every 20 samples. The LLCS is spiked at the Reporting Limit (RL)

9.4. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be prepared with every process batch of similar matrix, not to exceed twenty (20) samples. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

Note: Samples identified as field blanks, equipment blanks, or trip blanks should not be used for sample/sample duplicate nor MS/MSD analysis.

9.5. A duplicate control sample (LCS/LCSD) may be prepared when insufficient volume is provided to process a sample/sample duplicate or MS/MSD pair if required by client or program. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 of the Quality Control Program for further details.

10. CALIBRATION

10.1. A digestion vessel filled with reagent water is monitored for temperature for each digestion block. The analyst is to monitor this temperature for accuracy throughout the digestion and record the initial and final temperatures on the sample digestion log.

10.2. All air displacement pipettes must be verified over their range of use periodically as described in SOP WS-QA-0004.

10.3. All volumetric digestion vessels must be verified according to the frequency and procedures outlined in SOP WS-QA-0004. If the analyst suspects a vessel's reliability, a new verification of vessel may be required.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All preparation procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed. Double-check the sample bottle IDs, once prior to pouring them into the digestion vessels and before they are returned to the sample cart.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/plant) are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge-like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.6. Always read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.8. The following procedure must be followed for all aqueous sample preparations:

11.8.1. Use a small aliquot of each sample and measure the pH with pH indicator strips. If the bottles have an "X" on the tops, they have already been tested for other tests and do not require this step.

11.8.1.1. For samples with pH<2 put an "X" on top of the bottle indicating the samples were properly preserved. For these samples, record pH<2 on the preparation log.

11.8.1.2. For samples that test pH>2, notify the project manager immediately so they can contact the client. If the client requests the sample be preserved by the laboratory, add HNO_3 in 1.0 mL aliquots until the sample remains at pH<2 for at least 10 minutes. Let the sample stand for 24 hours and re-test the pH prior to preparation. As long as the pH<2, the sample can then be digested. A non-conformance memo must be created with an accounting of the anomalous event and the corrective action. Put an "X" on top of the bottle indicating the samples were properly preserved.

11.8.2. Mix the sample by shaking the container and pour the aliquot immediately. Shake the container between pouring the sample, MS, and SD.

11.8.3. Measure and transfer 50 mL of each sample into a graduated disposable digestion tube.

11.8.4. Measure two extra aliquots of the sample selected for the MS/MSD analysis.

11.8.5. Measure and transfer 50 mL of reagent water into digestion tubes for the method blank, LCS and LCSD (if required).

11.8.6. Using an adjustable volumetric air displacement pipette spike the LCS, LCSD, and MS/SD with the appropriate spiking solution/s shown in Tables I and II. ICP spikes require 0.5 mL of spiking solution and ICPMS spikes are spiked with 100 µL of spiking solution. A chemist trained in metals preparation and/or analysis must always witness the spiking procedure.

11.8.7. Using an adjustable volumetric air displacement pipette spike the LLCS with the appropriate spiking solution/s shown in Tables 1.1 and 2.2. ICP spikes require 0.5 mL of spiking solution and ICPMS spikes are spiked with 0.5 mL of spiking solution. A chemist trained in metals preparation and/or analysis must always witness the spiking procedure.

11.9. Proceed to the appropriate section for the desired method as follows:

Method 3005A, 200.8-5.4	11.10
Method 3010A, 200.7-4.4	11.11

11.10. Method 3005A - preparation for total recoverable or dissolved metals analysis by ICP or ICPMS (see Figure 1).

11.10.1. To each digestion tube, add 1 mL of concentrated HNO_3.

11.10.2. For ICP (method 6010B, 200.7-4.4) digests, add 2.5 mL of concentrated HCl. For ICPMS (method 6020, 200.8-5.4) digests, add 0.5 mL of concentrated HCl.

11.10.3. Heat at 90 - 95°C on a block digester until the volume is reduced to between 15 and 20 mL. Monitor the temperature regularly to assure proper heating.

Note: Do not allow samples to boil or go dry. Doing so will result in the loss of analytes and the samples must be re-prepared.

11.10.4. Cool the digestion tubes in a fume hood.

11.10.5. Adjust the final volume to 50 mL with reagent water. The sample is now ready for analysis

11.10.6. If insoluble material is present in the digestates, filter through Whatman 41 filter paper.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Warning: Filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic break or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustment when there are numerous samples to be processed.

Warning: The use of vacuum systems during filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

11.11. Method 3010A - preparation for total metals analysis by ICP (See Figure 2).

11.11.1. To the sample digestion tubes, add 1.5 mL of concentrated HNO_3.

11.11.2. Heat at approximately 95°C on block digesters until volume is reduced to between 15-20 mL. Monitor the temperature regularly to assure proper heating.

Note: Do not allow samples to boil or go dry. Doing so will result in the loss of analytes and the samples must be re-prepared.

11.11.3. Cool the digestion tubes in a fume hood to ambient temperature.

11.11.4. Add another1.5 mL portion of concentrated HNO_3.

11.11.5. Continue heating on the block digester for 20-25 minutes while ensuring that no portion of the bottom of the digestion tubes are allowed to go dry.

Note: If any sample shows a significant release of brown fumes while heating, add one more 1.5 mL aliquot of concentrated HNO_3 and heat for approximately 10 minutes.

11.11.6. Cool the digestion tubes in a fume hood to ambient temperature.

11.11.7. Add 2.5 mL of concentrated HCl.

11.11.8. Heat for an additional 15 minutes to dissolve precipitate or residue.

11.11.9. Cool the digestion tubes in a fume hood.

11.11.10. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.11.11. If insoluble material is present in the digestates, filter through Whatman 41 filter paper.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Warning: Filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic break or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustment when there are numerous samples to be processed.

Warning: The use of vacuum systems during filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked,

rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

12. CALCULATIONS/DATA REDUCTION

Not Applicable.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acidic waste generated by the extraction and unused acidic digestate containing nitric and hydrochloric acid. This acidic liquid is consolidated into a plastic acid waste drum. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Contaminated disposable materials such as plastic vials, pipettes, and filters used during sample preparation and digestion. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A, 3010A.

16.2. Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983, 1994. Methods 200.7, Revision 4.4, 1994; 200.8, Revision 5.4, 1994.

16.3. WS-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B and Method 200.7.

16.4. WS-MT-0001, Analysis of Metals by Inductively coupled Plasma/Mass Spectrometry.

17. METHOD MODIFICATIONS

17.1. Modifications applicable to SW-846 reference methods.

17.1.1. The totals sample volume has been reduced from 100 mL to 50 mL to accommodate hot block digestion.

17.2. Modifications Specific to Method 3005A

17.2.1. Method 3005A is being used for the preparation of “total recoverable” metals in drinking waters.

17.2.2. The amount of HCl added is reduced to 0.5 mL for ICPMS digests.

17.3. Modifications Specific to Method 3010A

17.3.1. Section 11.12.2 of this SOP requires the sample be reduced to a volume of 15 – 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the digestion tube should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the digestion tube so as to prevent the loss of critical analytes through volatilization.

17.3.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

17.4 Modifications Specific to Method 200.7-4.4

17.4.1 The initial sample volume has been reduced from 100 mL to 50 mL to accommodate hot block digestion.

17.4.2 The addition of 2 mL (1:1) nitric acid and 1.0 mL of (1:1) hydrochloric acid has been changed to 1 mL of nitric acid and 2.5 mL of hydrochloric acid.

17.5 Modifications Specific to Method 200.8-5.4

17.5.1 The initial sample volume has been reduced from 100 mL to 50 mL to accommodate hot block digestion.

17.5.2 The addition of 2 mL (1:1) nitric acid and 1.0 mL of (1:1) hydrochloric acid has been changed to 1 mL of nitric acid and 0.5 mL of hydrochloric acid.

18. ATTACHMENTS

18.1. Figure 1 — Method 3005A Flow Diagram

18.2. Figure 2 — Method 3010A Flow Diagram

18.3. Table 1 — Analyte List Certified Under NELAC Criteria: Method 6010B and Method 200.7

18.4. Table 1.1 — Analyte List Certified under NELAC Criteria: Method 6010B – Low Level Control Sample (LLCS)

18.5. Table 2 — Analyte List Certified Under NELAC Criteria: Method 6020 and Method 200.8

18.6. Table 2.1 — Analyte List Certified Under NELAC Criteria: Method 6020 - Low Level Control Sample (LLCS)

18.7. Appendix A — Contamination Control Guidelines

19. REVISION HISTORY

19.1. WS-IP-0001, Revision 5.4, Effective 12/12/2014

19.1.1. Inserted Section 2.3 – "Method 200.7-4.4 and Method 200.8-5.4 are applicable to the preparation of total recoverable metals by ICP and ICPMS. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to the original volume.

19.1.2. Inserted Section 17.4 – "Modifications Specific to Method 200.7-4.4:"

19.1.3. Inserted Section 17.5 – "Modifications Specific to Method 200.8-5.4"

19.1.4. Editorial changes.

19.2. WS-IP-0001, Revision 5.3, Effective 03/28/2014

19.2.1. Inserted Section 9.3.1 - For methods 6010B and 6020A a Low Level Control Sample (LLCS) must be prepared for every 20 samples.

19.2.2. Appended Section 9.3.1 – "The LLCS is spiked at the Reporting Limit (RL)"

19.2.3. Section 11.8.8.2, removed from the paragraph, "Record "AF" for "Anomaly Filed" on the digestion log."

19.2.4. Inserted Section 11.8.7 – "Using an adjustable volumetric air displacement pipette spike the LLCS with the appropriate spiking solution/s shown in Tables 1.1 and 2.2. ICP spikes require 0.5 mL of spiking solution and ICPMS spikes are spiked with 0.5 mL of spiking solution. A chemist trained in metals preparation and/or analysis must always witness the spiking procedure."

19.2.5. Inserted Tables 1.1 and 2.1.

19.2.6. Editorial changes.

19.3. WS-IP-0001, Revision 5.2, Effective 10/31/2012

19.3.1. Removed all references to Methods 200.7 and 200.8 from SOP.

19.3.2. Editorial changes.

19.4. WS-IP-0001, Revision 5.1, Effective 10/02/2009

19.4.1. Changed Section 11.8.6 "… ICPMS spikes are spiked with 1.0 mL…" to "…ICPMS spikes are spiked with 100 µL".

19.4.2. Changed "Table 2 - Analyte List Certified Under NELAC Criteria, Method 6020" Source from Spex XCAL-45 to Spec XCAL-58 and each of the Initial Concentrations except Hg are increased by a factor of 10x.

19.5. WS-IP-0001, Revision 5, Effective 09/15/2008

19.5.1. Updated to TestAmerica format.

Figure 1
Method 3005A

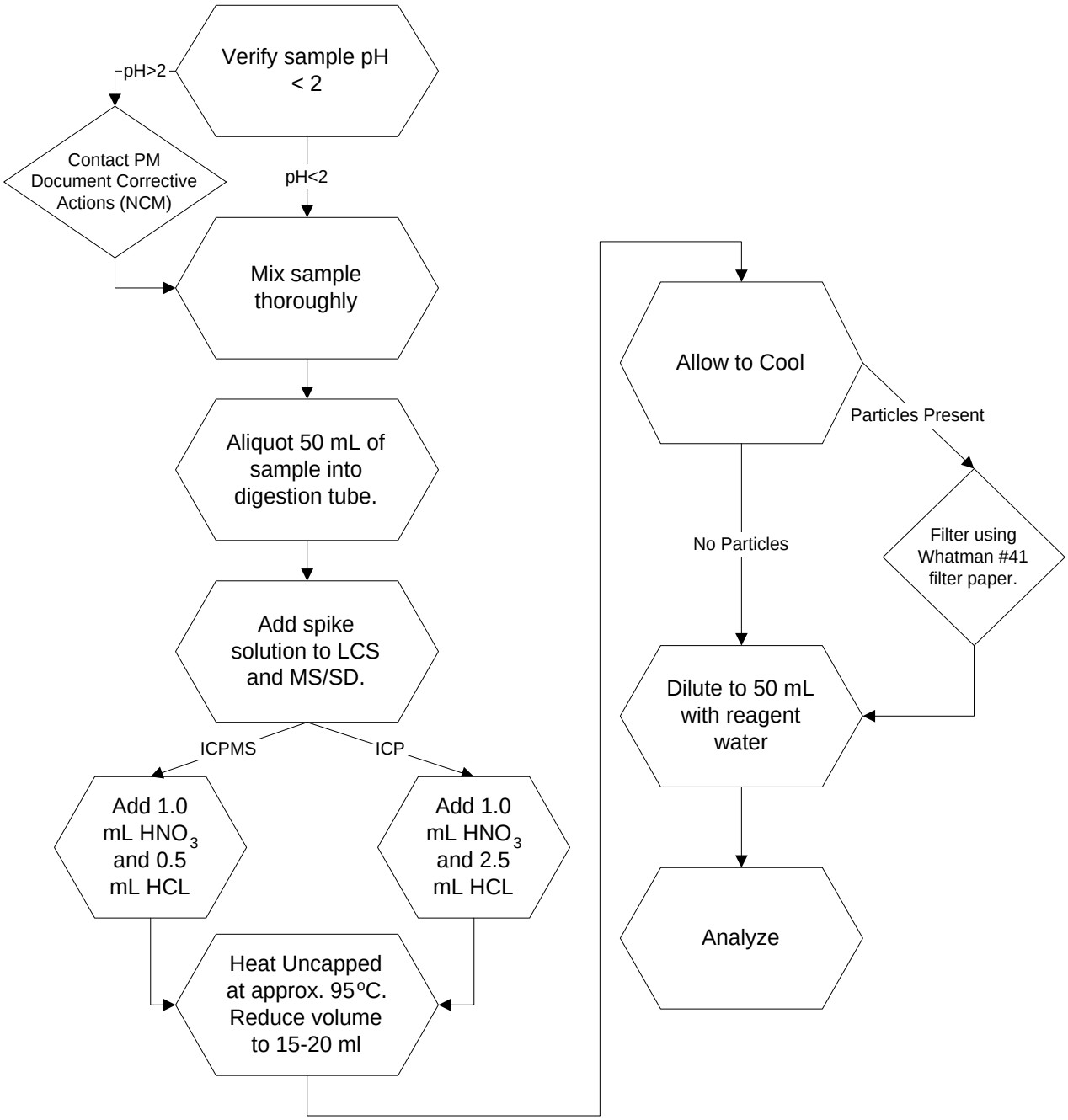

Figure 2

Method 3010A

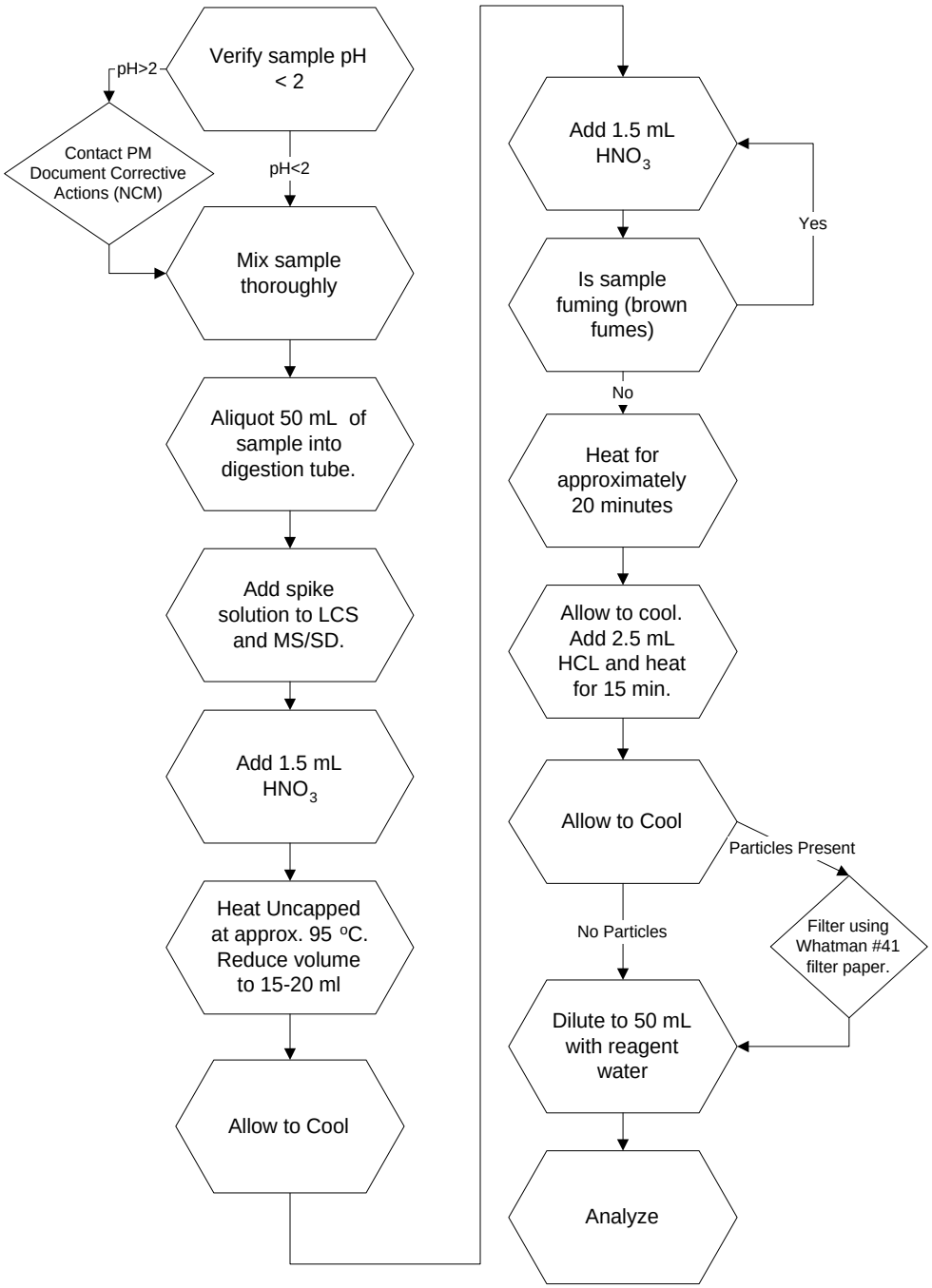

TABLE 1.

Analyte List Certified Under NELAC Criteria: Method 6010B & Method 200.7

Element	Symbol	Initial Conc mg/L	Final Conc mg/L	Source
Aluminum	Al	200	2.0	6010-SPIKE-1
Antimony	Sb	50	0.5	6010-SPIKE-2
Arsenic	As	200	2.0	6010-SPIKE-1
Barium	Ba	200	2.0	6010-SPIKE-1
Beryllium	Be	5.0	0.05	6010-SPIKE-1
Boron	B	100	1.0	6010-SPIKE-1
Cadmium	Cd	5.0	0.05	6010-SPIKE-1
Calcium	Ca	5000	50	6010-SPIKE-1
Chromium	Cr	20	0.2	6010-SPIKE-1
Cobalt	Co	50	0.5	6010-SPIKE-1
Copper	Cu	25	0.25	6010-SPIKE-1
Iron	Fe	100	1.0	6010-SPIKE-1
Lead	Pb	50	0.5	6010-SPIKE-1
Lithium	Li	100	1.0	6010-SPIKE-1
Magnesium	Mg	5000	50	6010-SPIKE-1
Manganese	Mn	50	0.5	6010-SPIKE-1
Molybdenum	Mo	100	1.0	6010-SPIKE-2
Nickel	Ni	50	0.5	6010-SPIKE-1
Phosphorous	P	1000	10	6010-SPIKE-1
Potassium	K	5000	50	6010-SPIKE-1
Selenium	Se	200	2.0	6010-SPIKE-1
Silicon	Si	1000	10	Inorganic Ventures
Silver	Ag	5	0.05	6010-SPIKE-2
Sodium	Na	5000	50	6010-SPIKE-1
Strontium	Sr	100	1.0	6010-SPIKE-1
Sulfur	S	1000	10	6010-SPIKE-2
Thallium	Tl	200	2.0	6010-SPIKE-1
Tin	Sn	200	2.0	6010-SPIKE-2
Titanium	Ti	100	1.0	6010-SPIKE-2
Vanadium	V	50	0.5	6010-SPIKE-1
Zinc	Zn	50	0.5	6010-SPIKE-1

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE 1.1 Analyte List Certified Under NELAC Criteria: Method 6010B.

Low Lever Control Sample (LLCS)

Element	Symbol	Initial Conc mg/L	Final Conc mg/L	Source
Aluminum	Al	0.5	0.005	Intermediate
Antimony	Sb	20	0.2	Intermediate
Arsenic	As	4	0.04	Intermediate
Barium	Ba	2	0.02	Intermediate
Beryllium	Be	0.3	0.003	Intermediate
Boron	B	20	0.2	Intermediate
Cadmium	Cd	0.3	0.003	Intermediate
Calcium	Ca	50	0.5	Intermediate
Chromium	Cr	1	0.01	Intermediate
Cobalt	Co	1	0.01	Intermediate
Copper	Cu	1.5	0.015	Intermediate
Iron	Fe	10	0.1	Intermediate
Lead	Pb	1	0.01	Intermediate
Lithium	Li	2	0.02	Intermediate
Magnesium	Mg	50	0.5	Intermediate
Manganese	Mn	1	0.01	Intermediate
Molybdenum	Mo	2	0.02	Intermediate
Nickel	Ni	1	0.01	Intermediate
Phosphorous	P	50	0.5	Intermediate
Potassium	K	100	1	Intermediate
Selenium	Se	4	0.04	Intermediate
Silicon	Si	50	0.5	Intermediate
Silver	Ag	0.5	0.005	Intermediate
Sodium	Na	50	0.5	Intermediate
Strontium	Sr	5	0.05	Intermediate
Sulfur	S	5	0.05	Intermediate
Thallium	Tl	3	0.03	Intermediate
Tin	Sn	2	0.02	Intermediate
Titanium	Ti	1	0.01	Intermediate
Vanadium	V	2	0.02	Intermediate
Zinc	Zn	2	0.02	Intermediate

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met

TABLE 2

Analyte List Certified Under NELAC Criteria: Method 6020 & Method 200.8

Element		Symbol		Source
		Initial Conc mg/L	Final Conc mg/L	
Aluminum	Al	500	1.0	TACA-1
Antimony	Sb	25	0.05	TACA-2
Arsenic	As	100	0.2	TACA-1
Barium	Ba	100	0.2	TACA-1
Beryllium	Be	100	0.2	TACA-1
Boron	B	500	1.0	TACA-1
Cadmium	Cd	100	0.2	TACA-1
Calcium	Ca	500	1.0	TACA-1
Chromium	Cr	100	0.2	TACA-1
Cobalt	Co	100	0.2	TACA-1
Copper	Cu	100	0.2	TACA-1
Iron	Fe	500	1.0	TACA-1
Lead	Pb	100	0.2	TACA-1
Lithium	Li	100	0.2	TACA-1
Magnesium	Mg	500	1.0	TACA-1
Manganese	Mn	100	0.2	TACA-1
Mercury	Hg	1.0	0.005	Inorganic Ventures
Molybdenum	Mo	100	0.2	TACA-2
Nickel	Ni	100	0.2	TACA-1
Phosphorous	P	500	1.0	TACA-1
Potassium	K	500	1.0	TACA-1
Selenium	Se	100	0.2	TACA-1
Silver	Ag	25	0 .05	TACA-1
Sodium	Na	500	1.0	TACA-1
Strontium	Sr	100	0.2	TACA-1
Thallium	Tl	25	0.05	TACA-1
Tin	Sn	100	0.2	TACA-2
Titanium	Ti	100	0.2	TACA-2
Uranium	U	100	0.2	TACA-1
Vanadium	V	100	0.2	TACA-1
Zinc	Zn	100	0.2	TACA-1

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE 2.1 — Analyte List Certified Under NELAC Criteria: Method 6020.

Low Level Control Sample (LLCS)

Element		Symbol		Source
		Initial Conc mg/L	Final Conc mg/L	
Aluminum	Al	10	0.1	Intermediate
Antimony	Sb	0.6	0.006	Intermediate
Arsenic	As	0.3	0.003	Intermediate
Barium	Ba	0.15	0.0015	Intermediate
Beryllium	Be	0.1	0.001	Intermediate
Boron	B	5	0.05	Intermediate
Cadmium	Cd	0.15	0.0015	Intermediate
Calcium	Ca	10	0.1	Intermediate
Chromium	Cr	0.5	0.005	Intermediate
Cobalt	Co	0.3	0.003	Intermediate
Copper	Cu	0.3	0.003	Intermediate
Iron	Fe	10	0.1	Intermediate
Lead	Pb	0.2	0.002	Intermediate
Lithium	Li	1	0.01	Intermediate
Magnesium	Mg	10	0.1	Intermediate
Manganese	Mn	0.6	0.006	Intermediate
Mercury	Hg	0.002	0.00002	Intermediate
Molybdenum	Mo	0.3	0.003	Intermediate
Nickel	Ni	0.3	0.003	Intermediate
Phosphorous	P	5	0.05	Intermediate
Potassium	K	10	0.1	Intermediate
Selenium	Se	0.3	0.003	Intermediate
Silver	Ag	0.1	0.001	Intermediate
Sodium	Na	10	0.1	Intermediate
Strontium	Sr	2	0.02	Intermediate
Thallium	Tl	0.15	0.0015	Intermediate
Tin	Sn	1	0.01	Intermediate
Titanium	Ti	0.5	0.005	Intermediate
Uranium	U	0.1	0.001	Intermediate
Vanadium	V	1.2	0.012	Intermediate
Zinc	Zn	1.2	0.012	Intermediate

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

APPENDIX A.

CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water. Disposable lab ware should be used whenever possible.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: Semivolatile Organic Compounds (Base/Neutral and Acids) by Gas Chromatography/Mass Spectrometry (GC/MS)

[Methods 8270C, 8270D, 625, and TO-13A]

Approvals (Signature/Date):

Victoria Nihart — Date Technical Manager	Joe Schairer — Date Health & Safety Manager / Coordinator
Lisa Stafford — Date Interim Quality Assurance Manager	Robert Hrabak — Date Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This method is based upon methods SW846 8270C, SW846 8270D, EPA Method 625, and TO-13A, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid, aqueous, and air matrices. Method 625 is applicable to the determination of the concentration of semivolatile compounds in extracts prepared from municipal and industrial wastewater. Direct injection of a sample may be used in limited applications. Refer to Table 1 for the list of compounds applicable for this method. Additional compounds may be amenable to this method. Refer to Section 4 for problematic analytes. If non-standard analytes are required, they must be validated by the procedures described in Section 13 before sample analysis.

1.2. The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Table 1 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.3. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Aqueous samples are extracted with methylene chloride using a separatory funnel, or a continuous extractor. Solid samples are extracted with methylene chloride/acetone using either sonication, or Soxhlet extraction. Waste dilution is used for samples that are miscible with the solvent. Air filter and absorbent cartridges are extracted by Soxhlet. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in WS-OP-0001 for aqueous, soil and waste, and in WS-OP-0006 for air matrices.

2.2. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3. DEFINITIONS

3.1. CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCCs is specified for calibration acceptance for Method 8270C.

3.2. SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance for Method 8270C.

3.3. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.4. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2. The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4. Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

4.5. The following compounds may require special treatment when being determined by this method:

4.5.1. Benzidine analytes can be subject to oxidative losses during solvent concentration and exhibits poor chromatography.

4.5.2. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

4.5.3. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.

4.5.4. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

4.5.5. 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method, and is reported as 4-methylphenol.

4.6. Hexachlorophene, kepone, famphur, and methapyrilene are unstable in methylene chloride and degrade soon after exposure to the solvent. Although these compounds can be detected in newly prepared standards and recently extracted samples, they cannot be detected in older calibration standards, and older standards are unable to meet calibration criteria for these four compounds.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the EHSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns or Requirements

5.1.1. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.1.2. The autovials used on the instrument contain a small amount of solvent after the analysis is completed, which can evaporate into the air through the puncture in the septa. These autovials may be collected in an open tripour or similar cup at the instrument while the analyst is removing them from the tray, but once that is complete the vials must be transferred to a waste container in a fume hood. They cannot be left out on the bench by the instrument.

5.1.3. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when working with solvent or extract containers. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.4. Exposure to chemicals must be maintained **as low as reasonably achievable**. All preparation of standards and dilutions shall be performed inside an operating fume hood. All samples must be opened, transferred and prepared

in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2. Gas Chromatograph/Mass Spectrometer System:

6.2.1. Agilent Technologies 6890N GC: Temperature-programmable for split/splitless injection.

6.2.2. Agilent Technologies 5975 Mass Selective Detector (MSD): Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

6.2.2.1. The mass spectrometer is capable of producing a mass spectrum for Decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 3 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.3. Column:

6.3.1. HP-5MS, 30 m x 0.25 mm I.D. (or 0.25 mm I.D.) 0.25-µm film thickness silicon-coated fused-silica capillary column.

6.3.1.1. The column is interfaced directly to the source.

6.3.1.2. Alternate columns are acceptable if they provide acceptable performance.

6.4. Data System:

6.4.1. Acquisition software: ChemStation Software version D.

6.4.1.1. The system provides the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.4.2. Data Processing Software: Chrom version. 2.1

6.4.2.1. The software can search any GC/MS data file for ions of a specific mass and can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). .

6.4.2.2. The software allows integrating the abundances in any EICP between specified time or scan-number limits.

6.4.2.3. The most recent version of the NIST Mass Spectral Library is recommended.

6.5. Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.6. Carrier gas: Ultra high purity helium.

7. REAGENTS AND STANDARDS

7.1. A minimum five-point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Tables 9 and 10 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2. An Internal Standard solution is prepared in methylene chloride (DCM). Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10. See Table 7 for composition of this solution. This solution is valid for up to one year.

7.2.1. Internal Standards are added to all standards and extracts to result in 40 ng injected onto the column. For example, if the volume of an extract used is 1000 µL, 20 µL of a 2000 µg/mL internal standard solution would be added for a 1 µL injection.

7.3. Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table

7.4. GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL o-decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 50µg/mL. This solution is valid for one year.

7.5. Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Table 5.

7.6. Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.7. The standards listed in 7.1 to 7.6 should be refrigerated at ≤ 6ºC when not in use. Refrigeration at -10ºC to -20ºC may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The continuing calibration standard is stored at ≤ 6ºC.

7.8. The daily instrument standard should be refreshed or reprepared on a weekly basis, to avoid concentration effects.

7.9. The standards must be replaced at least once a year. Rotate them to the Hazardous Waste storage area for disposal.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Refer to SOP WS-OP-0001 and WS-OP-0006 for sample bottle preservation and storage.

8.2. Extracts are stored at –10 to -20°C until at least 30 days following invoicing.

8.3. Extracts must be analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery ±3 standard deviations for surrogates, MS and LCS. Precision limits for matrix spikes/matrix spike duplicates are mean relative percent difference ± 3 standard deviations.

9.2.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is 5X or greater.

9.2.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into the LIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3. Refer to WS-PQA-003 for further details of control limits.

9.2.4. Current control limits for surrogates, LCS, and matrix spikes may be obtained by using QC Browser or an equivalent tool to query the LIMS database.

9.3. Method Blank

A method blank is prepared and analyzed with each batch of samples. The laboratory will attempt to run all samples from a preparation batch within the same sequence on the instrument. If this is not possible, samples may be run on different sequences or different instruments, if accompanied by an instrument blank as per Section 9.4. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP WS-OP-0001 and WS-OP-0006 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.
- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.
- The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low, and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.
- The DOD QSM criteria for method blanks is no analytes detected greater than ½ the reporting limit (RL). For common laboratory contaminants, it is no analytes detected greater than the RL.

9.3.1. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.2. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.3. Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4. Instrument Blank

Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of

methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5. Laboratory Control Sample (LCS)

A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS contains a representative subset of the analytes of interest (see Table 5), and must contain the same analytes as the matrix spike. Upon client request, additional analytes may be added to the LCS. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch. The analyst should monitor the system if non-control analytes are outside of established control limits, and perform corrective action as necessary to prevent re-occurring failures.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that sample was non-detect and the LCS had high recoveries, indicating a high bias).
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.
- If a sample is re-extracted past holding time, both sets of data will be reported.

9.5.1. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.5.2. A specific number of random exceedences may be allowed based on control charts kept by QA.

9.5.3. Poor performing analytes are those analytes identified as not performing well. These compounds generally have low percent recoveries and high standard deviations when the results of the LCS are compiled. Specific problems may also be noted by the standard manufacturers. The poor performing analytes for Method 8270 are aniline, 4-chloroaniline, 3-nitroaniline, 3,3'-dichlorobenzidine, benzoic acid, benzidine, and the "Appendix IX" compounds (see Table 1). Batch control is not determined by these analytes, and they are considered non-control analytes.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Table 5). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

Method 625 requires a matrix spike on 5% of the samples from each site being monitored. Compare the percent recovery (Ps) with the limits in Table 12.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the matrix spike/spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed if required by client or program. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Surrogates

9.7.1. Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 6.

9.7.2. Corrective actions for surrogate failures in MB, LCS, or LCSD (batch QC samples)

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Reanalyze the QC sample and/or reanalyze all samples in the QC batch.

Note: Unless otherwise specified by the client, it may be possible to report qualified results if method QC surrogate recoveries are biased high and analytes were not detected in the field samples. However, all other QC requirements would have to be met and the failure would have to be noted as an NCM.

9.7.3. Corrective actions for surrogate failures in field samples or MS/MSD:

- Check calculation and instrument performance.

- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses);
- consult with the client in the event of low bias (as evidenced by low recoveries) in conjunction with possible matrix effect;
- re-analyze or re-prepare the affected samples;
- and document the failure and note it in the final report

Note: Some client programs require reanalysis to confirm matrix interferences. Check special project instructions for this corrective action.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to re-prepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.7.4. If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.5. If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.6. If the reanalysis does confirm the original results, the sample will be re-extracted. If the re-extraction confirms, the original analysis is reported and the data flagged as estimated due to matrix effect. If re-extraction does not confirm (all surrogates pass in the re-extraction), only the re-extraction is reported (unless the re-extraction is outside holding times, in which case both sets of results are reported.

9.8. Internal Standards

Internal standards (IS) are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard response is monitored to verify that instrument performance is acceptable. Criteria for standards are delineated in Section 10 and must be met before sample analysis can start.

Internal standards in samples are monitored for instrument drift. If the internal standard data indicate instrument failure, the samples may require reanalysis. If IS response is affected by matrix effects, the impact on sample data is evaluated and the data is flagged appropriately.

9.8.1. Any samples that do not meet the same internal standard criteria set forth for the continuing calibration (Section 10.5) as compared to the Initial Calibration (ICAL) must be evaluated for validity. If multiple calibration mixtures are used, all IS are compared back to the primary ("HSL") calibration mixture. Alternatively, the internal standards in the samples may be compared to the response of the daily CCV, on a per batch basis, to meet client or program requirements. Evaluation may include re-analysis, unless there is obvious matrix interference. If the change in internal standard response is a matrix effect confined to an individual sample (and not due to instrument drift) reanalysis may not be necessary, but should be verified by the department manager or client. Any internal standard responses outside the criteria should be noted in the case narrative. If the change in internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.9. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.11. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 2.

10.3. All standards and extracts are allowed to warm to room temperature before injecting.

10.4. Instrument Tuning

At the beginning of each day (Method 625) or every twelve-hour shift (Method 8270) when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 3) are achieved for DFTPP (decafluorotriphenylphosphine).

10.4.1. Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectrum of DFTPP and confirm that all the key m/z criteria in Table 3 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.4.1.1. Evaluate the mass spectrum for DFTPP either at the apex of the peak, or ± 1 scan from the apex. Alternatively, average the mass spectra from 1 scan before the apex to 1 scan following the apex.

10.4.2. The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system.

10.4.2.1. Benzidine and pentachlorophenol should not exhibit excessive tailing. Tailing must be <3.0 for benzidine and < 5 for pentachlorophenol. Refer to Section 12 for the appropriate calculations.

10.4.2.2. Evaluation of tailing factor for 8270D must be ≤ 2 for benzidine and pentachlorophenol.

10.4.2.3. DDT must be included in the tuning standard, and its breakdown must be < 20%. Refer to Section 12 for the appropriate calculations.

10.5. Initial Calibration

10.5.1. Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards, as noted in Table 4. If interferences are noted, use one of the next two most intense masses for quantitation.

10.5.2. Compounds should be assigned to the IS with the closest retention time.

10.5.3. Prepare calibration standards to generate a multi-point curve for each parameter of interest. A minimum of five standards is required to generate a multi-point calibration quantitated based on average relative response factors

or linear regression. A minimum of six standards is required to generate a multi-point calibration based on non-linear curve fits (quadratic). In general, five standards are used for selected compounds known to exhibit poor response or poor chromatography at low concentrations, and all six standards are used for the remaining compounds. Compounds and the required calibration standards are identified in Tables 9 and 10.

10.5.4. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12 and verify that the minimum response factor and linearity criteria in Section 10.5.5 through 10.5.7 are met. **No sample analysis may be performed unless these criteria are met.**

10.5.5. Minimum Response Factor Criteria

10.5.5.1. Method 8270C utilizes System Performance Check Compounds (SPCCs) to demonstrate that the GCMS system is sufficiently inert to perform analysis. The minimum average RRF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. The following are the Method 8270C SPCCs:

- n-nitroso-di-n-propylamine
- Hexachlorocyclopentadiene
- 2,4-Dinitrophenol
- 4-Nitrophenol

10.5.5.2. For Method 8270D, compare the response factors for each calibration to minimums in Table 4. All analytes requiring minimum response factors in the initial calibration must exceed their required value in each level of the calibration curve. Any individual analyte that fails the minimum response factor must have a demonstration of sensitivity (low level CCV at or below the reporting limit) in each analytical batch to demonstrate sensitivity. The analyte must be detected and meet all standard qualitative identification criteria listed in Section 12. This low-level CCV is analyzed following the mid-level CCV.

10.5.6. CCC Criteria (Method 8270C)

Method 8270C contains separate linearity criteria for the Calibration Check Compounds (CCCs) and for non-CCCs. The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

If none of the CCCs are required analytes, evaluate the calibration curve per Section 10.4.7, or project specific calibration specifications must be agreed with the client. The CCCs are:

Phenol	Fluoranthene
Acenaphthene	Hexachlorobutadiene
1,4-Dichlorobenzene	Di-n-octylphthalate
n-nitrosodiphenylamine	4-Chloro-3-methylphenol
2-Nitrophenol	Benzo(a)pyrene
Pentachlorophenol	2,4,6-Trichlorophenol
2,4-Dichlorophenol	

10.5.7. Evaluate the %RSD of each analyte. For method 8270C, the RSD criteria is less than or equal to 15%. For Method 8270D, the RSD criteria is less than or equal to 20%. For method 625, RSD < 35% is acceptable.

10.5.7.1. If the %RSD is within the criteria above for a compound, the average relative response factor is used to quantitate that compound.

10.5.7.2. If the %RSD is greater than the criteria for a compound, attempt a linear curve fit. The linear calibration model may employ weighting such as 1/concentration^2 or 1/concentration (see also Section □). Method 8270D also permits forcing through zero to improve the fit at the lower end of the calibration curve. No matter what weighting factors or forcing modes are employed, linear curve fits must have a correlation coefficient (r) of ≥ 0.995, or a coefficient of determination (r^2) of ≥ 0.99. In addition, the y intercept (must be within ±1/2(RL/ISTD). If the intercept exceeds this criterion, false positives or negatives could result. If the RL is elevated for client or project requirements, use the default RL in the calculation.

Note: The RL value must be in terms of the amount "on-column" on the instrument.

10.5.7.3. If the linear fit does not meet criteria, a second-order ("quadratic curve") fit may be attempted. . The second order calibration model may employ weighting such as 1/concentration^2 or 1/concentration

(see also Section □). Method 8270D also permits forcing through zero to improve the fit at the lower end of the calibration curve. No matter what weighting factors or forcing modes are employed, the coefficient of determination (r^2) must be ≥ 0.990 and the y intercept) must be within ±1/2(RL/ISTD). If the intercept exceeds this criterion, false positives or negatives could result. If the RL is elevated for client or project requirements, use the default RL in the calculation

Note: The RL value must be in terms of the amount "on-column" on the instrument.

10.5.8. Additional Requirements for Method 8270D

- If more than 10% of the analytes fail both the 20% RSD and the 0.990 correlation coefficient, the initial calibration must be repeated.
- Any individual analyte that fails both the 20% RSD and the 0.990 correlation coefficient must have any positive result flagged as estimated or noted in an NCM. This option is not to be employed on a routine basis.
- Non-detect results for any analyte that fails both 20% RSD and 0.990 correlation coefficient may be reported without flagging if and only if there has been a successful analysis of a CCV at the reporting limit in the same analytical batch. The analyte must be detected and meet all standard qualitative identification criteria listed in Section 12.
- In general, no more than one or two of the poorest performing analytes should fail both 20% RSD and 0.990 correlation coefficient.

10.5.9. Whichever calibration model is used (average RRF or a linear or quadratic line fit), it is recommended that the calibration data be quantitated based on the calibration factors, ie, compare the calculated value of each calibration standard for each analyte against the expected amount. In general, the percent different of these read-backs should not exceed the calibration verification standard acceptance criteria (Section 10.6),

10.5.9.1. Method 8270D indicates that linear calibration models have a greater potential for significant bias at the lower portion of the calibration curve. Consequently, the read-back value for the lowest calibration level should be within ± 30% of the true concentration for each analyte in the standard.

10.5.10. Weighting of Data Points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental

analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.5.11. Initial calibration verification standard (ICV): When available, a second source calibration standard is analyzed with the initial calibration curve. Each compound must meet the %D criteria specified below.

- Method 8270C: Within ± 25%D.
- Method 8270D & Method 625: Within ± 30%D
- Permitted exceptions to the above criteria are listed below:

Classification	Criteria
Benzidine Analytes and Benzoic Acid:	Within ± 50%D
2,2'-Oxybis (1-chloropropane):	Within ± 30%D
Appendix IX:	Within ± 35%D for the following: a,a-Dimethylphenethylamine Acetophenone 2-Acetylaminofluorene 4-Aminobiphenyl Aramite Benzaldehyde Biphenyl Chlorobenzilate 1-Chloronaphthalene Diallate Dibenz(a,j)acridine 2,6-Dichlorophenol Dimethoate p-Dimethylaminoazobenzene 7,12-Dimethylbenz(a)anthracene 3,3'-Dimethylbenzidine 1,4-Dinitrobenzene Dinoseb Disulfoton Ethyl methanesulfonate Famphur 4 Hexachloropropene Isodrin Isosafrole Kepone Methapyrilene Methyl parathion 1,4-Naphthoquinone 1-Naphthylamine 2-Naphthylamine 4-Nitroquinoline-1-oxide n-Nitrosodi-n-butylamine n-Nitrosodiethylamine n-Nitrosodiphenylamine n-Nitrosomethylethylamine n-Nitrosomorpholine n-Nitrosopiperidine n-Nitrosopyrrolidine 5-Nitro-o-toluidine Parathion Pentachlorobenzene Pentachloroethane Pentachloronitrobenzene Phenacetin 1,4-Phenylenediamine Phorate 2-Picoline Pronamide Safrole Sulfotepp 2,3,5,6-Tetrachlorophenol Thionazin

Classification	Criteria	
	3-Methylcholanthrene Methyl methanesulfonate 1-Methylnaphthalene	o-Toluidine 4 O,O,O-Triethyl phosphorothioate 1,3,5-Trinitrobenzene

Some programs may specify different criteria, such as ≤ ± 25 %D for all analytes for AFCEE or DOD. Check QAPP requirements, or ensure that the client is aware of exceptions.

Under the DOD QSM, the criteria is to be within ± 20%D. The laboratory requests that the following compounds be permitted to meet 30%D when accepting QSM work, if these compounds are included in the requested analyte list:

Benzidine	1,3,5-Trinitrobenzene
Aniline	Dimethoate
3,3'-Dimethylbenzidine	2-Sec-Butyl-4,6-Dinitrophenol
p-Phenylenediamine	Methyl Parathion
1,2-Dinitrobenzene	4-Nitroquinoline
1,3-Dintrobenzene	Methapyrilene
1,4-Dinitrobenzene	Kepone
2,3,4,6-Tetrachlorophenol	Famphur

Corrective actions for a failed ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate instrument conditions.
- Evaluated the initial calibration standards.
- Narrate all affected data

10.5.12. If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to Section 10.5.

10.5.13. Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration

10.6. Continuing Calibration

10.6.1. At the beginning of each day (Method 625) or the start of each 12-hour period (Method 8270), the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 3.

10.6.1.1. Method 625 requires the tailing factor for Benzidine and Pentachlorophenol to be evaluated before sample analysis. The

benzidine tailing factor must be < 3.0 and the pentachlorophenol tailing factor must be < 5.

10.6.2. Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration. Note: use the 20 ng, 50 ng, 80 ng, or 120ng standard for the mid level standard. Whichever level is chosen, analyst must ensure that all quality control comparisons in 10.5.3 are measured are against the same level in the Initial Calibration.

10.6.3. Calculate the RRF for each compound in the standard.

10.6.3.1. For Method 8270C, the SPCC compounds (Section 10.5.5.1) must have a response factor of ≥ 0.05.

10.6.3.2. For Method 8270D, compare the response factors to the minimums in Table 4.

10.6.3.3. If the minimum response factor criteria are not met, the system should be evaluated and corrective action taken prior to sample analysis. Possible issues include degradation of the standard mixture and/or contaminated or active sites within the instrument.

10.6.4. Calculate the %D from the initial calibration for each compound (see Section 12).

10.6.4.1. For method 8270C, the %D for the CCC compounds must be within ± 20%. The %D for non-CCC compounds must be within ± 50%. If CCCs are not included in the list of compounds requested by the client, all compounds of interested for that client must have the %D within ± 20%.

10.6.4.2. For AFCEE, non-CCC compounds may not exceed ± 20%.

10.6.4.3. For DOD QSM work, the %D for each compounds of concern must be within ± 20%, unless the compound has been granted an exception in the client QAPP. The laboratory requests that the following compounds be permitted to meet 30%D when accepting QSM work, if these compounds are included in the requested analyte list.

Benzidine	1,3,5-Trinitrobenzene
Aniline	Dimethoate
3,3'-Dimethylbenzidine	2-Sec-Butyl-4,6-Dinitrophenol
p-Phenylenediamine	Methyl Parathion
1,2-Dinitrobenzene	4-Nitroquinoline
1,3-Dintrobenzene	Methapyrilene

1,4-Dinitrobenzene	Kepone
2,3,4,6-Tetrachlorophenol	Famphur

10.6.4.4. Method 8270D requires that the %D for each target compound be within ± 20%. Any sample detections for analytes that fail the CCV criteria must be flagged as estimated or described in an NCM. Whenever possible, samples should be reanalyzed with a passing CCV. Samples that are non-detect for analytes that fail the CCV criteria must have a low-level CCV (at or below the reporting limit) in the analytical batch as a sensitivity demonstration. The low-level CCV must meet all criteria for qualitative identification.

10.6.4.5. Method 625 requires that the %D for each target analyte to be within ± 20%.

10.6.4.6. If the %D criteria above are not met, the system must be evaluated and corrective action must be taken. The DFTPP tune and continuing calibration must be acceptable before analysis begins.

10.6.5. The internal standard response must be within 50-200% of the response in the same concentration level of the initial calibration.

10.6.6. The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration, or retention times are updated

10.6.7. Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample injected less than 12 hours after the DFTPP is acceptable.).

11. PROCEDURE

11.1. Procedural Variations

One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.2. Sample Preparation

Samples are prepared following SOP WS-OP-0001 or WS-OP-0006.

11.3. Sample Analysis Procedure

11.3.1. Calibrate the instrument as described in Section 10. Depending on the target compounds required by the client, it might be necessary to use more than one calibration standard.

11.3.2. All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

11.3.3. Add internal standard to the extract to result in 40 ng injected on column (for example, 20 µL internal standard solution in 1.0 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

11.3.4. Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.3.5. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.3.6. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See CA-Q-S-002 for Manual Integration procedures.

11.3.7. Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.3.8. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in Section 12.3.

11.4. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be targeted to be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the reporting limit and the matrix allows for analysis at a lesser dilution, the sample should be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.4.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.4.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4.3. Each time the sample extract is diluted, internal standard solution must be added to maintain a final concentration of 40 µg/mL for each internal standard component. See Table 8 for guidance as to solution volumes when diluting.

11.5. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at -10 to -20°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.6. Retention Time Criteria for Samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.6.1. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.7. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP WS-OP-0013 for determination of percent moisture.

11.8. Troubleshooting Guide

11.8.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the Sacramento Quality Assurance Manual (QAM), the following daily maintenance should be performed.

- Clip column as necessary.

- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

11.8.2. Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2. Mass Chromatogram Searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compounds hexachlorophene, kepone and famphur

fall into this category, and are required for Appendix IX analysis. For these analytes a mass chromatogram search is made.

12.2.1. Display the mass chromatograms for the masses below for the region of the chromatogram from at least 3 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for all ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene, kepone, and/or famphur. No quantitation is possible.

Compound	Masses Monitored		
Hexachlorophene	196	198	
Kepone	272	274	237
Famphur	218	125	93

12.3. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.
- Any compound identified by the library search with a probability of 80% or less will be reported as UNKNOWN only. Any compound identified by the library search with 80% to 100% probability will be reported as the compound selected by the library search program. The only exception will be Aldol Condensate

products which will be reported as "Aldol Condensate Product", and not by the analyte name.

Note: For samples that request a specific analyte list, and a TIC evaluation, any analytes detected that are not on the specific analyte list, but are part of laboratory calibration, are to be reported from the calibration, not library search.

12.4. Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Mass	Analytes	Mass	Analytes
93	Aniline/bis(2-Chloroethyl)ether	142, 141, 115	2- and 1-Methylnaphthalene
146, 148, 111	1,3-, 1,4-, 1,2-Dichlorobenzene	202, 101, 100	Fluoranthene and Pyrene
196, 198, 200	2,4,6- and 2,4,5-Trichlorophenol	228, 229, 226	Benzo(a)anthracene and Chrysene
232, 230, 131	2,3,5,6- and 2,3,4,6-Tetrachlorophenol	252, 253, 125	Benzo(b)fluoranthene and Benzo(k)fluoranthene
178, 176, 179	Phenanthrene and Anthracene	276	Indeno(1,2,3-cd)pyrene and Benzo(g,h,i)perylene
108, 107, 79	3/4 Methylphenol & 2-Methylphenol	65, 92, 138	2-, 3-, and 4- nitroaniline
162, 127, 164	1- and 2- chloronaphthalene	165, 63, 89	2,4- and 2,6-Dinitrotoluene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times. In particular, Benzo(b) and (k)fluoranthene may present difficulty in separation at higher levels. They may be reported together if the client does not require the separation.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

a,a-Dimethylphenethylamine
Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations is appropriate for these compounds.

12.5. Calculations

More detailed information on fitting calculations is available from CA-Q-S-005 Calibration Curves (General).

12.5.1. Percent Relative Standard Deviation for Initial Calibration

EQUATION 1

$$\%\text{RSD} = \frac{\text{SD}}{\overline{\text{RF}}} \times 100$$

Where:

$\overline{RF}$ = Mean of RFs from initial calibration for a compound,

SD = Standard Deviation of RFs from the initial calibration for that compound, calculated as:

EQUATION 2

$$\sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

Where:

RFi = RF for each of the calibration levels

N = Number of RF levels

12.5.2. Continuing Calibration Percent Drift

EQUATION 3

$$\%\text{Drift} = \frac{C_{found} - C_{actual}}{C_{actual}} \times 100\%$$

Where:

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3. Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration. Variables for the equations below include:

C_{ex} = Concentration in extract, µg/mL
R_x = Response for analyte
R_{is} = Response for internal standard
C_{is} = Concentration of internal standard
A = Intercept
B = Slope
C = Curvature

12.5.3.1. Average Response Factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤15%, the average response factor from the initial calibration may be used for quantitation.

EQUATION 4

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

12.5.3.2. Linear Fit

EQUATION 5

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

12.5.3.3. Quadratic Fit

EQUATION 6

$$C_{ex} = \left(\frac{\frac{R_x}{R_{is}} - A}{B}\right)(C_{is})$$

12.5.4. The concentration in the sample is then calculated.

12.5.4.1. Aqueous Calculation

EQUATION 7

$$\text{Concentration, } \mu g/L = \frac{CexVt}{Vo}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)
V_o = Volume of water extracted (mL)

12.5.4.2. Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

EQUATION 8

$$\text{Concentration, } \mu g/kg = \frac{CexVt}{WsD}$$

Where:

W_s = Weight of sample extracted or diluted in grams
D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.5.5. MS/MSD Percent Recovery Calculation.

EQUATION 9

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result
S_R = Sample result
S_A = Spike added

12.5.6. Relative Percent Difference calculation for the MS/MSD

EQUATION 10

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

12.5.7. Relative Response Factor Calculation.

EQUATION 11

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured (µg/L)
C_{is} = Concentration of the specific internal standard (µg/L)

12.5.8. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x= Area of the total ion chromatogram for the compound being measured
A_{is}= Area of the total ion chromatogram for the nearest internal standard without interference
RF= 1

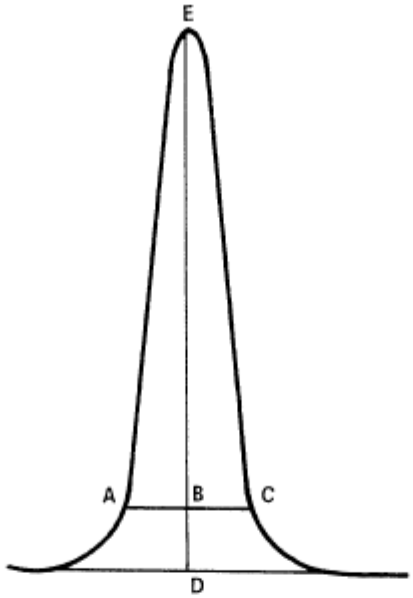
12.5.9. Percent DDT Breakdown

EQUATION 12

$$\text{\% DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

12.5.10. Tailing Factor (From Method 625)



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

EQUATION 13

$$\text{Tailing Factor} = \frac{AB}{BC}$$

Where:

AB = Peak width at 10% peak height, leading line to line drawn from apex.
BC = Peak width at 10% peak height, line from apex to right side of peak.

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: CA-Q-S-006 and WS-QA-0006. MDLs are available in the Quality Assurance department.

13.2. Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.1.1. For Method 625 the concentration of the QC check sample is 100 ug/L.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.2.2.1. For Method 625 the standard deviation and the recovery for each analyte of interest is compare to (s) and (P) in Table 12

13.2.3. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Non-standard Analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.5. Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.1. Do not allow waste solvent to evaporate in fume hoods. All solvent waste is stored in capped containers unless transfers are being made.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

15.1. Autovials contaminated with methylene chloride. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methylene chloride to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Determinative Chromatographic Separations, Method 8000B.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, March 2003, Determinative Chromatographic Separations, Method 8000C.

16.4. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update IV, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270D, Revision 4, February 2007.

16.5. J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975).

16.6. Method 625, 40 CRF Part 136, Appendix A. (March 2014)

16.7. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-13A, (1999)

17. METHOD MODIFICATIONS

17.1. Modifications from Reference Method (8270C and 820D)

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2. The quantitation and qualifier ions for some compounds have been changed

from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.3. Due to retention time shifts as a result of column clipping and long-lived initial calibrations, the internal standard retention time criteria for continuing calibrations has been removed.

18. ATTACHMENTS

18.1. Table 1- Analytes and Standard Reporting Limits

18.2. Table 2- Suggested Instrument Conditions

18.3. Table 3- DFTPP Key Ions and Ion Abundance Criteria

18.4. Table 4- Analytes in Appropriate Retention Time Order and Characteristic Ions

18.5. Table 5- LCS and MS/MSD Spiking Components and Concentrations in the Sample

18.6. Table 6- Surrogate Compounds and Spiking Levels

18.7. Table 7- Internal Standard Solution

18.8. Table 8- Dilution Guidance

18.9. Table 9- Calibration Levels, Primary Standard, µg/mL

18.10. Table 10- Calibration Levels, Appendix IX Standard, µg/mL

18.11. Table 11 - Summary of EPA Method 625 Quality Control

18.12. Table 12 – QC Acceptance Criteria -Method 625

18.13. Table 17 – Method Accuracy and Precision as Functions of Concentration – Method 625

19. REVISION HISTORY

19.1. WS-MS-0005, Revision 5.0, Effective 04/15/2014

19.1.1. Changed title of SOP to “Semivolatile Organic Compounds (Base/Neutral and Acids) by Gas Chromatography/Mass Spectrometry [Methods 8270C, 8270D, 625, and TP-13A”.

19.1.2. Added language in section 1.1 for Method 625 is applicable to municipal and industrial wastewater.

19.1.3. Inserted section 9.6 to state Method 625 requires a matrix spike on 5% of the samples from each site being monitored.

19.1.4. Language added in section 10.4 that the instrument tuning is performed at the beginning of each day for Method 625.

19.1.5. Criteria clarified in section 10.4.2.1 for the benzidine tailing factor to be < 3.0 and the tailing factor to be < 5 for pentachlorophenol.

19.1.6. Sections 10.5.5.2 and 10.6.4.4 expanded, and Section 10.5.8 added to include language from the Corporate Quality Policy Memorandum CA-Q-QM-009.

19.1.7. Section 10.5.7, appended, "For method 625, RSD < 35% is acceptable."

19.1.8. Section 10.6 was updated for the DFTPP to be analyzed at the beginning of each day for Method 625 and added tailing factor criteria for benzidine and pentachlorophenol.

19.1.9. Inserted section 10.6.4.5 for Method 625 CCV criteria is ± 20% of the predicted response.

19.1.10. Inserted image of tailing factor calculation in section 12.5.10.

19.1.11. Inserted section 13.2.1.1 for the concentration of the QC check sample concentration for Method 625 is 100 ug/L.

19.1.12. Inserted section 13.2.2.1 for Method 625 QC check sample standard deviation (s) and percent recovery (P) is compared to (s) and (P) in Table 12.

19.1.13. Changed the reference date for Method 625 to 3/31/2014.

19.1.14. Added Table 11 – Summary of Method 625 Quality Control.

19.1.15. Added Table 12 – QC Acceptance Criteria Method 625.

19.2. WS-MS-0005, Revision 4.5, Effective 07/15/2013

19.2.1. Added Reference to Methods 8270D and TO-13A in the title.

19.2.2. Added criteria for Method 8270D in Sections 10.5 and 10.6.

19.2.3. Reconciled excepted analytes for QSM for section 10.5 and 10.6 with the current exceptions document.

19.2.4. Added masses to the commonly confused compounds in Section 12.4.

19.2.5. Added 8270D minimum RRFs to Table 4.

19.3. WS-MS-0005, Revision 4.4, Effective 06/25/2013

19.3.1. Section 6.2: Changed column type used for analyses.

19.3.2. Section 10.5.7.3: Removed reference to forcing the curve through zero.

19.3.3. Editorial changes.

19.4. WS-MS-0005, Revision 4.3, Effective 8/9/2010

19.4.1. Changed Section 10.6.3 to: "The % drift for all non-CCC and non-SPCC analytes must be <50% (20% AFCEE or DOD, or predetermined exceptions).

19.4.2. Inserted not following Section 12.5: More detailed information on fitting calculations are available from CA-Q-S-005 Calibration Curves (General).

Table 1				
Analytes and Standard Reporting Limits				
Compound	CAS No.	Aqueous ug/L	Solid ug/kg	Air ug/sample
a,a-Dimethylphenethylamine [4]	122-09-8	50	1600	50
Acenaphthene	83-32-9	10	330	10
Acenaphthylene	208-96-8	10	330	10
Acetophenone [4]	98-86-2	10	330	10
2-Acetylaminofluorene [4]	53-96-3	100	3300	100
4-Aminobiphenyl [4]	92-67-1	50	1600	50
Aniline	62-53-3	10	330	10
Anthracene	120-12-7	10	330	10
Aramite [4]	140-57-8	20	660	20
Azobenzene[2]	103-33-3	10	330	10
Benzaldehyde [4]	100-52-7	10	330	--
Benzidine	92-87-5	100	3300	100
Benzo(a)anthracene	56-55-3	10	330	10
Benzo(b)fluoranthene	205-99-2	10	330	10
Benzo(j)fluoranthene	205-82-3	10	330	10
Benzo(k)fluoranthene	207-08-9	10	330	--
Benzoic acid	65-85-0	50	1600	150
Benzo(ghi)perylene	191-24-2	10	330	10
Benzo(a)pyrene	50-32-8	10	330	10
Benzo(e)pyrene	197-97-2	10	330	10
Benzyl alcohol	100-51-6	10	330	10
Biphenyl [4]	92-52-4	10	330	--
bis(2-Chloroethoxy)methane	111-91-1	10	330	10
bis(2-Chloroethyl) ether	111-44-4	10	330	10
bis(2-Ethylhexyl) phthalate	117-81-7	10	330	10
4-Bromophenyl phenyl ether	101-55-3	10	330	10
Butyl benzyl phthalate	85-68-7	10	330	10
Carbazole	86-74-8	10	330	10
4-Chloroaniline	106-47-8	10	330	10
Chlorobenzilate [4]	510-15-6	20	660	20
4-Chloro-3-methylphenol	59-50-7	10	330	50
1-Chloronaphthalene [4]	90-13-1	10	330	--
2-Chloronaphthalene	91-58-7	10	330	10
2-Chlorophenol	95-57-8	10	330	10
4-Chlorophenyl phenyl ether	7005-72-3	10	330	10
Chrysene	218-01-9	10	330	10
Diallate [4]	2303-16-4	20	660	20
Dibenz(a,j)acridine [4]	224-42-0	20	660	20
Dibenz(a,h)anthracene	53-70-3	10	330	10

Table 1				
Analytes and Standard Reporting Limits				
Compound	CAS No.	Aqueous ug/L	Solid ug/kg	Air ug/sample
Dibenzofuran	132-64-9	10	330	10
Di-n-butyl phthalate	84-74-2	10	330	10
1,2-Dichlorobenzene	95-50-1	10	330	10
1,3-Dichlorobenzene	541-73-1	10	330	10
1,4-Dichlorobenzene	106-46-7	10	330	10
3,3'-Dichlorobenzidine	91-94-1	50	1600	10
2,4-Dichlorophenol	120-83-2	10	330	10
2,6-Dichlorophenol [4]	87-65-0	10	330	10
Diethyl phthalate	84-66-2	10	330	10
Dimethoate [4]	60-51-5	20	660	20
p-Dimethylaminoazobenzene [4]	60-11-7	20	660	20
7,12-Dimethylbenz(a)anthracene [4]	57-97-6	20	660	20
3,3'-Dimethylbenzidine [4]	119-93-7	50	1600	50
2,4-Dimethylphenol	105-67-9	10	330	10
Dimethyl phthalate	131-11-3	10	330	10
1,3-Dinitrobenzene	99-65-0	10	330	10
1,4-Dinitrobenzene [4]	100-25-4	10	330	--
4,6-Dinitro-2-methylphenol	534-52-1	50	1600	50
2,4-Dinitrophenol	51-28-5	50	1600	50
2,4-Dinitrotoluene	121-14-2	10	330	10
2,6-Dinitrotoluene	606-20-2	10	330	10
Dinoseb [4]	88-85-7	20	660	20
Di-n-octyl phthalate	117-84-0	10	330	10
Disulfoton [4]	298-04-4	50	1600	50
Ethyl methanesulfonate [4]	62-50-0	10	330	10
Famphur [4]	52-85-7	100	3300	100
Fluoranthene	206-44-0	10	330	10
Fluorene	86-73-7	10	330	10
Hexachlorobenzene	118-74-1	10	330	10
Hexachlorobutadiene	87-68-3	10	330	10
Hexachlorocyclopentadiene	77-47-4	50	1600	50
Hexachloroethane	67-72-1	10	330	10
Hexachloropropene [4]	1888-71-7	10	3300	10
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10
Isodrin [4]	465-73-6	20	660	20
Isophorone	78-59-1	10	330	10
Isosafrole [4]	120-58-1	20	660	20
Kepone [4]	143-50-0	100	3300	100
Methapyrilene [4]	91-80-5	50	1600	50

Table 1				
Analytes and Standard Reporting Limits				
Compound	CAS No.	Aqueous ug/L	Solid ug/kg	Air ug/sample
3-Methylcholanthrene [4]	56-49-5	20	660	20
Methyl methanesulfonate [4]	66-27-3	10	330	10
2-Methylnaphthalene	91-57-6	10	330	10
1-Methylnaphthalene [4]	90-12-0	10	330	--
Methyl parathion [4]	298-00-0	50	1600	50
2-Methylphenol	95-48-7	10	330	20
3-Methylphenol [4]	108-39-4	10	330	50
4-Methylphenol	106-44-5	10	330	20
3-Methylphenol & 4-Methylphenol	65794-96-9	20	660	50
Naphthalene	91-20-3	10	330	10
1,4-Naphthoquinone [4]	130-15-4	50	1600	50
1-Naphthylamine [4]	134-32-7	10	330	10
2-Naphthylamine [4]	91-59-8	10	330	10
2-Nitroaniline	88-74-4	50	1600	10
3-Nitroaniline	99-09-2	50	1600	10
4-Nitroaniline	100-01-6	50	1600	50
Nitrobenzene	98-95-3	10	330	10
2-Nitrophenol	88-75-5	10	330	50
4-Nitrophenol	100-02-7	50	1600	50
4-Nitroquinoline-1-oxide [4]	56-57-5	100	3300	100
n-Nitrosodi-n-butylamine [4]	924-16-3	10	330	10
n-Nitrosodiethylamine [4]	55-18-5	10	330	10
n-Nitrosodimethylamine	62-75-9	10	330	10
n-Nitrosodiphenylamine [3,4]	86-30-6	10	330	10
n-Nitrosodi-n-propylamine	621-64-7	10	330	10
n-Nitrosomethylethylamine [4]	10595-95-6	10	330	10
n-Nitrosomorpholine [4]	59-89-2	10	330	10
n-Nitrosopiperidine [4]	100-75-4	10	330	10
n-Nitrosopyrrolidine [4]	930-55-2	10	330	10
5-Nitro-o-toluidine [4]	99-55-8	20	660	20
2,2'-oxybis(1-Chloropropane)[1]	108-60-1	10	330	10
Parathion [4]	56-38-2	50	1600	50
Pentachlorobenzene [4]	608-93-5	20	660	20
Pentachloroethane [4]	76-01-7	50	1600	50
Pentachloronitrobenzene [4]	82-68-8	50	1600	50
Pentachlorophenol	87-86-5	50	1600	50
Phenacetin [4]	62-44-2	20	660	20
Phenanthrene	85-01-8	10	330	10
Phenol	108-95-2	10	330	50

Table 1				
Analytes and Standard Reporting Limits				
Compound	CAS No.	Aqueous ug/L	Solid ug/kg	Air ug/sample
1,4-Phenylenediamine [4]	106-50-3	100	3300	100
Phorate [4]	298-02-2	50	1600	50
2-Picoline [4]	109-06-8	20	660	20
Pronamide [4]	23950-58-5	20	660	20
Pyrene	129-00-0	10	330	10
Pyridine	110-86-1	20	660	20
Safrole [4]	94-59-7	20	660	20
Sulfotepp [4]	3689-24-5	50	1600	50
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	10
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600	50
2,3,5,6-Tetrachlorophenol [4]	935-95-5	50	1600	--
Thionazin [4]	297-97-2	50	1600	50
o-Toluidine [4]	95-53-4	20	660	20
1,2,4-Trichlorobenzene	120-82-1	10	330	10
2,4,5-Trichlorophenol	95-95-4	10	330	10
2,4,6-Trichlorophenol	88-06-2	10	330	50
O,O,O-Triethyl phosphorothioate [4]	126-68-1	50	1600	50
1,3,5-Trinitrobenzene [4]	99-35-4	50	1600	50

[1] 2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

[3] Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Note: Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See Section 12.2.1)

[4] Appendix IX compounds

Table 2	
Suggested Instrumental Conditions	
Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	60°C for 1 minute
Column Temperature Program	Ramp at 20 °C/min to 190 °C, hold for 0.00 min Ramp at 13 °C/min to 310 °C, hold for 2.00 min. Ramp at 30 °C/min to 320 °C, hold for 1.00 min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split/splitless
Sample Volume	1 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 3
DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 4					
Analytes in Approximate Retention Time Order and Characteristic Ions					
	Approximate Retention Time	Primary ion	Secondary ion	Tertiary ion	8270D Min RRF
Internal Standards					
1,4-Dichlorobenzene-d4	4.045	152	150	115	
Naphthalene-d8	5.432	136	68		
Acenaphthene-d10	7.512	164	162	160	
Phenanthrene-d10	9.457	188	94	80	
Chrysene-d12	13.855	240	120	236	
Perylene-d12	16.245	264	260	265	
Surrogates					
2-Fluorophenol	2.907	112	64		
Phenol-d5	3.735	99	71		
2-Chlorophenol-d4	3.859	132	68	134	
1,2-Dichlorobenzene-d4	4.242	152	150		
Nitrobenzene-d5	4.666	82	128	54	
2-Fluorobiphenyl	6.725	172	171	170	
2,4,6-Tribromophenol	8.536	330	332	141	
Terphenyl-d14	12.065	244	122	212	
Analytes					
n-Nitrosodimethylamine[1,2]	2.006	74	42	44	0.050
Pyridine[3]	2.017	79	78		0.050
2-Picoline	2.433	93	66	92	0.050
n-Nitrosomethylethylamine[2]	2.516	88	35		0.050
Methyl methanesulfonate	2.754	80	79	65	0.050
Benzaldehyde	3.582	106	105	77	0.010
n-Nitrosodiethylamine	3.064	102	56	57	0.050
Ethyl methanesulfonate	3.323	79	109	97	0.050
Aniline	3.776	93	66	65	0.050
Phenol	3.755	94	65	66	0.800
Pentachloroethane	3.706	117	119		0.050
bis(2-chloroethyl)ether	3.828	93	63	95	0.700
2-Chlorophenol	3.879	128	64	130	0.800
1,3-Dichlorobenzene	4.014	146	148	111	0.050
1,4-Dichlorobenzene	4.066	146	148	111	0.050
Benzyl Alcohol	4.221	108	79	77	0.050
1,2-Dichlorobenzene	4.252	146	148	113	0.050
2-Methylphenol[1,2]	4.345	108	107	79	0.700
2,2'-oxybis(1-Chloropropane)	4.366	45	121	77	0.010
4-Methylphenol[1,2]	4.511	108	107	79	0.600
Acetophenone[2]	4.43	105	77	51	0.010
Hexachloroethane[2]	4.562	117	201	119	0.300
n-Nitrosodi-n-propylamine	4.542	70	42	130	0.500

Table 4					
Analytes in Approximate Retention Time Order and Characteristic Ions					
	Approximate Retention Time	Primary ion	Secondary ion	Tertiary ion	8270D Min RRF
n-Nitrosopyrrolidine	4.43	100	41	42	0.050
3-Methylphenol[1,2]	4.461	108	107	79	0.050
o-Toluidine	4.482	106	107	77	0.050
n-Nitrosomorpholine[3]	4.451	56	86	116	0.050
Nitrobenzene	4.687	77	123	65	0.200
n-Nitrosopiperidine[1,2]	4.782	42	114	55	0.050
Isophorone	4.945	82	95	138	0.400
2-Nitrophenol	5.028	139	109	65	0.100
2,4-Dimethyphenol	5.09	107	122	121	0.200
bis(2-chloroethoxy)methane	5.194	93	95	123	0.300
o,o,o-TEPT[3]	5.144	198	121	93	0.050
2,4-Dichlorophenol	5.297	162	164	98	0.200
Benzoic Acid	5.349	122	105	77	0.010
1,2,4-Trichlorobenzene	5.38	180	182	145	0.050
Naphthalene	5.452	128	129	127	0.700
a-,a-Dimethylphenethylamine	5.196	58	91	42	0.050
4-Chloroaniline	5.545	127	129		0.010
2,6-Dichlorophenol[3]	5.506	162	164	98	0.050
Hexachloropropene	5.537	213	211	215	0.050
Hexachlorobutadiene	5.659	225	223	227	0.010
p-Phenylenediamine[2]	5.91	108	80	107	0.050
n-Nitroso-di-n-butylamine	5.91	84	57	41	0.050
4-Chloro-3-methylphenol	6.125	107	144	142	0.200
Safrole[2]	6.117	162	131	104	0.050
Biphenyl[3]	6.769	154	153	76	0.010
2-Methylnaphthalene	6.249	142	141	115	0.400
1-Chloronaphthalene	6.81	162	127	164	0.050
1-Methylnaphthalene	6.324	142	141	115	0.050
Hexachlorocyclopentadiene	6.518	237	235	272	0.050
1,2,4,5-Tetrachlorobenzene	6.448	216	214	218	0.010
Isosafrole#1	6.469	162	131	104	0.050
2,4,6-Trichlorophenol	6.632	196	198	200	0.200
2,4,5-Trichlorphenol	6.673	196	198	200	0.200
2-Chloronaphthalene	6.829	162	127	164	0.800
Isosafrole#2	6.727	162	131	104	0.050
2-Nitroaniline	7.015	65	92	138	0.010
1,4-Naphthoquinone	6.996	158	102	104	0.050
1,3-Dinitrobenzene	7.1	168	75		0.050
Dimethylphthalate	7.274	163	194	164	0.010
Acenaphthylene	7.325	152	151	153	0.900
1,4-Dinitrobenzene	7.234	168	75		0.050

Table 4					
Analytes in Approximate Retention Time Order and Characteristic Ions					
	Approximate Retention Time	Primary ion	Secondary ion	Tertiary ion	8270D Min RRF
2,6-Dinitrotoluene	7.832	165	63	89	0.200
1,2-Dinitrobenzene	7.369	168	63	50	0.050
3-Nitroaniline	7.522	138	108	92	0.010
Acenaphthene[1,2]	7.553	153	154	152	0.050
2,4-Dinitrophenol	7.646	184	63	154	0.010
Dibenzofuran	7.75	168	139	84	0.800
4-Nitrophenol[1,2]	7.77	109	139	65	0.010
Pentachlorobenzene	7.721	250	252	248	0.050
2,4-Dinitrotoluene	8.184	165	63	89	0.200
1-Naphthylamine[2]	7.793	143	115	116	0.050
2,3,5,6-Tetrachlorophenol	7.855	232	230	131	0.050
2-Naphthylamine	7.897	143	115	116	0.050
2,3,4,6-Tetrachlorophenol[3]	7.917	232	230	131	0.010
Fluorene	8.184	166	165	167	0.900
Diethylphthalate[3]	8.164	149	177	150	0.010
4-Chlorophenyl phenyl ether	8.195	204	206	141	0.400
4-Nitroaniline[3]	8.329	138	108	92	0.010
5-Nitro-o-toluidine	8.207	152	106	77	0.050
Thionazin[1]	8.186	97	96	143	0.050
4,6-Dinitro-2-methylphenol[1,2]	8.37	198	182	77	0.010
n-Nitrosodiphenylamine	8.381	169	168	167	0.010
Diphenylamine	8.3	169	168	167	0.050
Azobenzene	8.402	77	105	182	0.050
4-Bromophenyl phenyl ether	8.836	248	250	141	0.100
Sulfotepp[3]	8.673	97	322	202	0.050
Diallate 1	8.745	86	234		0.010
1,3,5-Trinitrobenzene[1,2]	8.766	120	75	213	0.050
Phorate[1,2]	8.756	121	75	97	0.050
Diallate 2	8.859	86	234		0.010
Phenacetin	8.797	108	109	179	0.050
Hexachlorobenzene	9.033	284	142	249	0.100
Dimethoate	9.004	87	93	125	0.050
Pentachlorophenol	9.302	266	264	268	0.050
4-Aminobiphenyl	9.159	169	168	170	0.050
Pentachloronitrobenzene[1,2]	9.345	295	237	142	0.010
Pronamide	9.325	173	175	145	0.050
Phenanthrene	9.498	178	179	176	0.700
Anthracene	9.571	178	176	179	0.700
Disulfoton	9.501	88	97	89	0.050
2-sec-Butyl-4,6-dinitrophenol	9.532	211	163	147	0.050

Table 4					
Analytes in Approximate Retention Time Order and Characteristic Ions					
	Approximate Retention Time	Primary ion	Secondary ion	Tertiary ion	8270D Min RRF
Carbazole[3]	9.83	167	166	139	0.010
Methyl parathion	10.07	109	125	263	0.050
Di-n-butylphthalate	10.502	149	150	104	0.010
Hexachlorophene	10.6	196	198	406	0.050
4-Nitroquinoline-1-oxide[1,2]	10.68	190	174	101	0.050
Parathion	10.711	109	97	291	0.050
Methapyrilene[1,2]	10.877	58	97	72	0.050
Isodrin	11.063	193	66	195	0.050
Fluoranthene[2]	11.361	202	101	100	0.600
Benzidine	11.64	184	92	185	0.050
Pyrene[2]	11.734	202	101	100	0.600
Aramite#1[2]	12.057	185	135	91	0.010
Aramite#2[2]	12.191	185	91	135	0.010
p-Dimethylaminoazobenzene[1,2]	12.274	120	225	77	0.050
p-Chlorobenzilate	12.388	251	139	253	0.050
Kepone	12.802	272	274	237	0.050
3,3'-Dimethylbenzidine	12.924	212	106	196	0.050
Famphur	12.864	218	125	93	0.050
Butylbenzylphthalate	13.037	149	91	206	0.010
2-Acetylaminofluorene	13.329	181	180	223	0.050
Benzo(a)anthracene	13.834	228	229	226	0.800
Chrysene	13.917	228	226	229	0.700
3,3'-Dichlorobenzidine	13.876	252	254	126	0.010
bis(2-Ethylhexyl)phthalate	14.145	149	167	279	0.010
Di-n-octylphthalate	15.2	149	167	43	0.010
Benzo(j)fluoranthene	15.596	252	126	250	0.050
Benzo(b)fluoranthene	15.676	252	253	125	0.700
Benzo(k)fluoranthene	15.718	252	253	125	0.700
7,12-Dimethylbenz(a)anthracene[2]	15.606	256	241	257	0.050
Benzo(e)pyrene[3]	16.09	252	126	250	0.050
Benzo(a)pyrene	16.173	252	253	125	0.700
3-Methylcholanthrene[2]	16.651	268	253	267	0.050
Dibenz(a,j)acridine	17.531	279	280	277	0.050
Indeno(1,2,3-cd)pyrene	17.911	276	138		0.500
Dibenzo(a,h)anthracene	17.953	278	139	279	0.400
Benzo(g,h,i)perylene	18.356	276	138	277	0.500

1 Primary ion differs from Table 1 of Method 8270C (Characteristic Ions).

2 One of more confirmation ions differs from Table 1 of Method 8270C.

3 Compound is not listed in Table 1 of Method 8270C

Table 5			
LCS and MS/SD Spiking Components and Concentrations in the Sample			
Compound	Aqueous (µg/L)	Solid (µg/kg)	Air (µg)
Acenaphthene	100	3330	100
Acenaphthylene	100	3330	100
Anthracene	100	3330	100
Benz(a)anthracene	100	3330	100
Benzo(b)fluoranthene	100	3330	100
Benzo(k)fluoranthene	100	3330	100
Benzoic acid[1]	100	3330	100
Benzo(ghi)perylene	100	3330	100
Benzo(a)pyrene	100	3330	100
Benzyl alcohol	100	3330	100
bis(2-Chloroethoxy)methane	100	3330	100
bis(2-Chloroethyl) ether	100	3330	100
bis(2-Ethylhexyl) phthalate	100	3330	100
4-Bromophenyl phenyl ether	100	3330	100
Butyl benzyl phthalate	100	3330	100
Carbazole	100	3330	100
4-Chloroaniline[1]	100	3330	100
4-Chloro-3-methylphenol	100	3330	100
2-Chloronaphthalene	100	3330	100
2-Chlorophenol	100	3330	100
4-Chlorophenyl phenyl ether	100	3330	100
Chrysene	100	3330	100
Dibenz(a,h)anthracene	100	3330	100
Dibenzofuran	100	3330	100
Di-n-butyl phthalate	100	3330	100
1,2-Dichlorobenzene	100	3330	100
1,3-Dichlorobenzene	100	3330	100
1,4-Dichlorobenzene	100	3330	100
3,3'-Dichlorobenzidine[1]	100	3330	100
2,4-Dichlorophenol	100	3330	100
Diethylphthalate	100	3330	100
2,4-Dimethylphenol	100	3330	100
Dimethylphthalate	100	3330	100
4,6-Dinitro-2-methylphenol	100	3330	100
2,4-Dinitrophenol	100	3330	100
2,4-Dinitrotoluene	100	3330	100
2,6-Dinitrotoluene	100	3330	100
Di-n-octylphthalate	100	3330	100
Fluoranthene	100	3330	100

Table 5			
LCS and MS/SD Spiking Components and Concentrations in the Sample			
Compound	Aqueous (µg/L)	Solid (µg/kg)	Air (µg)
Fluorene	100	3330	100
Hexachlorobenzene	100	3330	100
Hexachlorobutadiene	100	3330	100
Hexachlorocyclopentadiene	100	3330	100
Hexachloroethane	100	3330	100
Indeno(1,2,3-cd)pyrene	100	3330	100
Isophorone	100	3330	100
2-Methylnaphthalene	100	3330	100
2-Methylphenol	100	3330	100
4-Methylphenol	200	6670	200
Naphthalene	100	3330	100
2-Nitroaniline	100	3330	100
3-Nitroaniline[1]	100	3330	100
4-Nitroaniline	100	3330	100
Nitrobenzene	100	3330	100
2-Nitrophenol	100	3330	100
4-Nitrophenol	100	3330	100
n-Nitrosodimethylamine	100	3330	100
n-Nitrosodiphenylamine	100	3330	100
n-Nitrosodi-n-propylamine	100	3330	100
2,2'-Oxybis(1-chloropropane)	100	3330	100
Pentachlorophenol	100	3330	100
Phenanthrene	100	3330	100
Phenol	100	3330	100
Pyrene	100	3330	100
1,2,4-Trichlorobenzene	100	3330	100
2,4,5-Trichlorophenol	100	3330	100
2,4,6-Trichlorophenol	100	3330	100

[1] These are poor performers.

Table 6			
Surrogate Compounds and Spiking Levels			
Compound	Aqueous ug/L	Solid ug/kg	Air ug
2-Fluorobiphenyl	50	1660	50
2-Fluorophenol	75	2500	100
2,4,6-Tribromophenol	75	2500	100
Nitrobenzene-d5	50	1660	50
Phenol-d5	75	2500	100
Terphenyl-d14	50	1660	50
2-Chlorophenol-d4	75	2500	100
1,2-Dichlorobenzene-d4[1]	50	1660	50

[1] For Air work, this compound is spiked onto the media during preparation, before the air sample is collected.

Table 7			
Internal Standard Solution			
Component	**Concentration in Solution µg/mL**	**Concentration in Extract µg/mL**	**ng on-column (1µL injection)**
1,4-Dichlorobenzene-d4	2000	40	40
Naphthalene-d8	2000	40	40
Acenaphthene-d10	2000	40	40
Phenanthrene-d10	2000	40	40
Chrysene-d12	2000	40	40
Perylene-d12	2000	40	40

Table 8			
Dilution Guidance			
Dilution Factor[1]	µL of Sample Extract	µL IS Solution	µL DCM
1x (undiluted)	1000	20	NA
2x	500	10	500
5x	200	16	800
10x	100	18	900
20x	50	19	950

[1] For further dilutions, make serial dilutions. In general, add 1 µL IS solution for every 50 µL DCM added to the extract. In all cases, the µL IS solution to be added assumes that the source solution (undiluted extract, previous extract dilution, etc) was already spiked with IS solution.

Table 9
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Pyridine	5	10	20	50	80	120	160
n-Nitrosodimethylamine	5	10	20	50	80	120	160
Aniline	5	10	20	50	80	120	160
Phenol	5	10	20	50	80	120	160
bis(2-Chloroethyl)ether	5	10	20	50	80	120	160
2-Chlorophenol	5	10	20	50	80	120	160
1,3-Dichlorobenzene	5	10	20	50	80	120	160
1,4-Dichlorobenzene	5	10	20	50	80	120	160
Benzyl alcohol	5	10	20	50	80	120	160
1,2-Dichlorobenzene	5	10	20	50	80	120	160
2-Methylphenol	5	10	20	50	80	120	160
2,2'-Oxybis(1-chloropropane)[1]	5	10	20	50	80	120	160
4-Methylphenol	5	10	20	50	80	120	160
n-Nitroso-di-n-propylamine	5	10	20	50	80	120	160
Hexachloroethane	5	10	20	50	80	120	160
Nitrobenzene	5	10	20	50	80	120	160
Isophorone	5	10	20	50	80	120	160
2-Nitrophenol	5	10	20	50	80	120	160
2,4-Dimethylphenol	5	10	20	50	80	120	160
Benzoic acid	5	10	20	50	80	120	160
bis(2-chloroethoxy)methane	5	10	20	50	80	120	160
2,4-Dichlorophenol	5	10	20	50	80	120	160
1,2,4-Trichlorobenzene	5	10	20	50	80	120	160
Naphthalene	5	10	20	50	80	120	160
4-Chloroaniline	5	10	20	50	80	120	160
Hexachlorobutadiene	5	10	20	50	80	120	160
4-Chloro-3-methylphenol	5	10	20	50	80	120	160
2-Methylnaphthalene	5	10	20	50	80	120	160
Hexachlorocyclopentadiene	5	10	20	50	80	120	160
2,4,6-Trichlorophenol	5	10	20	50	80	120	160
2,4,5-Trichlorophenol	5	10	20	50	80	120	160
2-Chloronaphthalene	5	10	20	50	80	120	160
2-Nitroaniline	5	10	20	50	80	120	160
Dimethylphthalate	5	10	20	50	80	120	160
Acenaphthylene	5	10	20	50	80	120	160
3-Nitroaniline	5	10	20	50	80	120	160
Acenaphthene	5	10	20	50	80	120	160
2,4-Dinitrophenol	5	10	20	50	80	120	160
4-Nitrophenol	5	10	20	50	80	120	160
Dibenzofuran	5	10	20	50	80	120	160
2,4-Dinitrotoluene	5	10	20	50	80	120	160
2,6-Dinitrotoluene	5	10	20	50	80	120	160
Diethylphthalate	5	10	20	50	80	120	160
4-Chlorophenyl phenyl ether	5	10	20	50	80	120	160
Fluorene	5	10	20	50	80	120	160

Table 9
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
4-Nitroaniline	5	10	20	50	80	120	160
4,6-Dinitro-2-methylphenol	5	10	20	50	80	120	160
n-Nitrosodiphenylamine	5	10	20	50	80	120	160
Azobenzene[2]	5	10	20	50	80	120	160
4-Bromophenyl phenyl ether	5	10	20	50	80	120	160
Hexachlorobenzene	5	10	20	50	80	120	160
Pentachlorophenol	5	10	20	50	80	120	160
Phenanthrene	5	10	20	50	80	120	160
Anthracene	5	10	20	50	80	120	160
Carbazole	5	10	20	50	80	120	160
Di-n-butylphthalate	5	10	20	50	80	120	160
Fluoranthene	5	10	20	50	80	120	160
Benzidine	5	10	50	100	80	120	160
Pyrene	5	10	20	50	80	120	160
Butylbenzylphthalate	5	10	20	50	80	120	160
3,3'-Dichlorobenzidine	5	10	20	50	80	120	160
Benzo(a)anthracene	5	10	20	50	80	120	160
bis(2-Ethylhexyl)phthalate	5	10	20	50	80	120	160
Chrysene	5	10	20	50	80	120	160
Di-n-octylphthalate	5	10	20	50	80	120	160
Benzo(b)fluoranthene	5	10	20	50	80	120	160
Benzo(k)fluoranthene	5	10	20	50	80	120	160
Benzo(a)pyrene	5	10	20	50	80	120	160
Indeno(1,2,3-cd)pyrene	5	10	20	50	80	120	160
Dibenz(a,h)anthracene	5	10	20	50	80	120	160
Benzo(g,h,i)perylene	5	10	20	50	80	120	160

Calibration Levels, Surrogate Standard, µg/mL

Analyte	Level 1	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
2-Fluorophenol	5	10	20	50	80	120	160
Phenol-d5	5	10	20	50	80	120	160
2-Chlorophenol-d4	5	10	20	50	80	120	160
1,2-Dichlorobenzene-d4	5	10	20	50	80	120	160
Nitrobenzene-d5	5	10	20	50	80	120	160
2-Fluorobiphenyl	5	10	20	50	80	120	160
2,4,6-Tribromophenol	5	10	20	50	80	120	160
Terphenyl-d14	5	10	20	50	80	120	160

[1] 2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether
[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 10
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
2-Picoline	10	20	50	80	120	160
n-Nitrosomethylethylamine	10	20	50	80	120	160
Methyl methanesulfonate	10	20	50	80	120	160
n-Nitrosodiethylamine	10	20	50	80	120	160
Ethyl methanesulfonate	10	20	50	80	120	160
Pentachloroethane	10-	20	50	80	120	160
Acetophenone	10	20	50	80	120	160
n-Nitrosopyrrolidine	10	20	50	80	120	160
n-Nitrosomorpholine	10	20	50	80	120	160
o-Toluidine	10-	20	50	80	120	160
3-Methylphenol	10	20	50	80	120	160
n-Nitrosopiperidine	10	20	50	80	120	160
o,o,o-Triethyl-Phosphorothioate	10-	20	50	80	120	160
a,a-Dimethylphenethylamine	10-	20	50	80	120	160
2,6-Dichlorophenol	10	20	50	80	120	160
Hexachloropropene	10-	20	50	80	120	160
p-Phenylenediamine	10-	20	50	80	120	160
n-Nitrosodi-n-butylamine	10	20	50	80	120	160
Safrole	10-	20	50	80	120	160
1,2,4,5-Tetrachlorobenzene	10	20	50	80	120	160
Isosafrole 1 + 2	10-	20	50	80	120	160
1,4-Dinitrobenzene	10	20	50	80	120	160
1,4-Naphthoquinone	10-	20	50	80	120	160
1,3-Dinitrobenzene	10	20	50	80	120	160
Pentachlorobenzene	10	20	50	80	120	160
1-Naphthylamine	10	20	50	80	120	160
2-Naphthylamine	10	20	50	80	120	160
2,3,4,6-Tetrachlorophenol	10-	20	50	80	120	160
5-Nitro-o-toluidine	10-	20	50	80	120	160
Thionazin	10-	20	50	80	120	160
1,3,5-Trinitrobenzene	10-	20	50	80	120	160
Sulfotepp	10-	20	50	80	120	160
Phorate	10-	20	50	80	120	160
Phenacetin	10-	20	50	80	120	160
Diallate 1 + 2	10-	20	50	80	120	160
Dimethoate	10	20	50	80	120	160
4-Aminobiphenyl	10-	20	50	80	120	160
Pentachloronitrobenzene	10-	20	50	80	120	160
Pronamide	10-	20	50	80	120	160
Disulfoton	10-	20	50	80	120	160
2-sec-Butyl-4,6-dinitrophenol (Dinoseb)	10-	20	50	80	120	160
Methyl parathion	10-	20	50	80	120	160
4-Nitroquinoline-1-oxide	10-	20	50	80	120	160

Table 10
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Parathion	10-	20	50	80	120	160
Isodrin	10	20	50	80	120	160
Kepone	10-	20	50	80	120	160
Famphur	10-	20	50	80	120	160
Methapyrilene	10-	20	50	80	120	160
Aramite 1 and 2	10-	20	50	80	120	160
p-(Dimethylamino)azobenzene	10-	20	50	80	120	160
p-Chlorobenzilate	10	20	50	80	120	160
3,3'-Dimethylbenzidine	10-	20	50	80	120	160
2-Acetylaminofluorene	10-	20	50	80	120	160
Dibenzo (a,j)acridine	10-	20	50	80	120	160
7,12-Dimethylbenz(a)anthracene	10-	20	50	80	120	160
3-Methylcholanthrene	10-	20	50	80	120	160

Table 11
Summary of EPA Method 625 Quality Control

Quality Control Objective	Frequency	Criteria	Corrective Action
Calibration curve (minimum of 3 points)	As needed prior to sample analysis	<35% RSD to use Ave .RF; Alternatively plot a calibration curve of response ratios, As/Ais, vs concentration ratios Cs/Cis	
Calibration Curve Verification (CCV)	Each working day	± 20% of the predicted response for each parameter	Repeat using a fresh calibration standard; If fails ± 20 % prepare and analyze a new calibration curve.
Initial Demonstration of Proficiency (IDOC)	Initial one-time demonstration	Must meet standard deviation (s) and average recovery (X), listed in Table 12, for each parameter.	For any parameter that fails to meet s or X, locate and correct the source the problem. Repeat the test for the parameters that fail.
Matrix Spike (MS)	5% of the samples from each sample site being monitored	Evaluate the percent recovery with (P) percent in Table 12	Analyze a QC check sample and calculate the percent recovery (Ps). Compare the (Ps) for each parameter that failed in the MS with Table 12. If the (Ps) is outside the acceptance criteria in Table 12 for that parameter the analytical result for that parameter in the unspikes sample is suspect and can not be reported for regulatory compliance purposes.
QC Check Sample	Should be routinely analyzed with the matrix spike sample	Compare the percent recoveries (Ps) in Table 12	Evaluate for those compounds that fail in the matrix spike.
GC/MS Performance Test	At the beginning of each day; 50ng DFTPP; 100ng Benzidine; 50ng Pentachlorophenol	Obtain background corrected mass spectra of DFTPP. Must meet key m/z criteria in Table 3; Benzidine tailing factor must be < 3.0; Pentachlorophenol tailing factor must be < 5.	Perform corrective action and repeat the GC/MS performance test.
Qualitative Identification	Evaluation of the EICP for the primary, secondary and tertiary masses for each parameter detected.	Characteristic masses maximize within one scan of each other; RT must fall within ± 30s of the RT of the standard; Ion abundances of the three characteristic masses in the EICP must be within ± 20% of the relative intensities if the reference spectrum.	The criteria must be met to make a qualitative identification.

Table 12
QC Acceptance Criteria—Method 625

Parameter	Test concentration (µg/L)	Limits for s (µg/L)	Range for X(µg/L)	Range for P, P_s (Percent)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benzo(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether[a]	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4′-DDD	100	31.0	D-134.5	D-145
4,4′-DDE	100	32.0	19.2-119.7	4-136
4,4′-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4,-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3′-Dhlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146

Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26-155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Dichlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s=Standard deviation for four recovery measurements, in µg/L (Section 8.2.4).
X=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

Table 13
Method Accuracy and Precision as Functions of Concentration—Method 625

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
Acenaphthene	0.96C=0.19	0.15X−0.12	0.21X−0.67
Acenaphthylene	0.89C=0.74	0.24X−1.06	0.26X−0.54
Aldrin	0.78C=1.66	0.27X−1.28	0.43X=1.13
Anthracene	0.80C=0.68	0.21X−0.32	0.27X−0.64
Benzo(a)anthracene	0.88C−0.60	0.15X=0.93	0.26X−0.28
Benzo(b)fluoranthene	0.93C−1.80	0.22X=0.43	0.29X=0.96
Benzo(k)fluoranthene	0.87C−1.56	0.19X=1.03	0.35X=0.40
Benzo(a)pyrene	0.90C−0.13	0.22X=0.48	0.32X=1.35
Benzo(ghi)perylene	0.98C−0.86	0.29X=2.40	0.51X−0.44
Benzyl butyl phthalate	0.66C−1.68	0.18X=0.94	0.53X=0.92
β-BHC	0.87C−0.94	0.20X−0.58	0.30X−1.94
δ-BHC	0.29C−1.09	0.34X=0.86	0.93X−0.17
Bis(2-chloroethyl) ether	0.86C−1.54	0.35X−0.99	0.35X=0.10
Bis(2-chloroethoxy)methane	1.12C−5.04	0.16X=1.34	0.26X=2.01
Bis(2-chloroisopropyl) ether[a]	1.03C−2.31	0.24X=0.28	0.25X=1.04
Bis(2-ethylhexyl) phthalate	0.84C−1.18	0.26X=0.73	0.36X=0.67
4-Bromophenyl phenyl ether	0.91C−1.34	0.13X=0.66	0.16X=0.66
2-Chloronaphthalene	0.89C=0.01	0.07X=0.52	0.13X=0.34
4-Chlorophenyl phenyl ether	0.91C=0.53	0.20X−0.94	0.30X−0.46
Chrysene	0.93C−1.00	0.28X=0.13	0.33X−0.09
4,4′-DDD	0.56C−0.40	0.29X−0.32	0.66X−0.96
4,4′-DDE	0.70C−0.54	0.26X−1.17	0.39X−1.04
4,4′-DDT	0.79C−3.28	0.42X=0.19	0.65X−0.58
Dibenzo(a,h)anthracene	0.88C=4.72	0.30X=8.51	0.59X=0.25
Di-n-butyl phthalate	0.59C=0.71	0.13X=1.16	0.39X=0.60
1,2-Dichlorobenzene	0.80C=0.28	0.20X=0.47	0.24X=0.39
1,3-Dichlorobenzene	0.86C−0.70	0.25X=0.68	0.41X=0.11
1,4-Dichlorobenzene	0.73C−1.47	0.24X=0.23	0.29X=0.36
3,3′-Dichlorobenzidine	1.23C−12.65	0.28X=7.33	0.47X=3.45
Dieldrin	0.82C−0.16	0.20X−0.16	0.26X−0.07
Diethyl phthalate	0.43C=1.00	0.28X=1.44	0.52X=0.22
Dimethyl phthalate	0.20C=1.03	0.54X=0.19	1.05X−0.92
2,4-Dinitrotoluene	0.92C−4.81	0.12X=1.06	0.21X=1.50
2,6-Dinitrotoluene	1.06C−3.60	0.14X=1.26	0.19X=0.35
Di-n-octyl phthalate	0.76C−0.79	0.21X=1.19	0.37X=1.19

Endosulfan sulfate	0.39C=0.41	0.12X=2.47	0.63X−1.03
Endrin aldehyde	0.76C−3.86	0.18X=3.91	0.73X−0.62
Fluoranthene	0.81C=1.10	0.22X−0.73	0.28X−0.60
Fluorene	0.90C−0.00	0.12X=0.26	0.13X=0.61
Heptachlor	0.87C−2.97	0.24X−0.56	0.50X−0.23
Heptachlor epoxide	0.92C−1.87	0.33X−0.46	0.28X=0.64
Hexachlorobenzene	0.74C=0.66	0.18X−0.10	0.43X−0.52
Hexachlorobutadiene	0.71C−1.01	0.19X=0.92	0.26X=0.49
Hexachloroethane	0.73C−0.83	0.17X=0.67	0.17X=0.80
Indeno(1,2,3-cd)pyrene	0.78C−3.10	0.29X=1.46	0.50X=0.44
Isophorone	1.12C=1.41	0.27X=0.77	0.33X=0.26
Naphthalene	0.76C=1.58	0.21X−0.41	0.30X−0.68
Nitrobenzene	1.09C−3.05	0.19X=0.92	0.27X=0.21
N-Nitrosodi-n-propylamine	1.12C−6.22	0.27X=0.68	0.44X=0.47
PCB-1260	0.81C−10.86	0.35X=3.61	0.43X=1.82
Phenanthrene	0.87C−0.06	0.12X=0.57	0.15X=0.25
Pyrene	0.84C−0.16	0.16X=0.06	0.15X=0.31
1,2,4-Trichlorobenzene	0.94C−0.79	0.15X=0.85	0.21X=0.39
4-Chloro-3-methylphenol	0.84C=0.35	0.23X=0.75	0.29X=1.31
2-Chlorophenol	0.78C=0.29	0.18X=1.46	0.28X=0.97
2,4-Dichlorophenol	0.87C=0.13	0.15X=1.25	0.21X=1.28
2,4-Dimethylphenol	0.71C=4.41	0.16X=1.21	0.22X=1.31
2,4-Dinitrophenol	0.81C−18.04	0.38X=2.36	0.42X=26.29
2-Methyl-4,6-Dinitrophenol	1.04C−28.04	0.05X=42.29	0.26X=23.10
2-Nitrophenol	1.07C−1.15	0.16X=1.94	0.27X=2.60
4-Nitrophenol	0.61C−1.22	0.38X=2.57	0.44X=3.24
Pentachlorophenol	0.93C=1.99	0.24X=3.03	0.30X=4.33
Phenol	0.43C=1.26	0.26X=0.73	0.35X=0.58
2,4,6-Trichlorophenol	0.91C−0.18	0.16X=2.22	0.22X=1.81

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
s_r′=Expected single analyst standard deviation of measurements at an average concentration found of X, in µg/L.
S′= Expected interlaboratory standard deviation of measurements at an average concentration found of X, in µg/L.
C= True value for the concentration, in µg/L.
X= Average recovery found for measurements of samples containing a concentration of C, in µg/L.
[a]The proper chemical name is 2,2′oxybis(1-chloropropane).

Title: Determination of Volatile Organics and Total Purgeable Petroleum Hydrocarbons by GC/MS

[Methods 8260B, 8260C, 624, CA-LUFT, and AK 101]

Approvals (Signature/Date):

Victoria Nihart 8/27/14	8/27/14
Victoria Nihart — Date	Joe Schairer — Date
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9/20/2014	8/27/14
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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, municipal and industrial discharges, soils, sludges and other solid matrices. Standard analytes and reporting limits are listed in Table 1. Total Purgeable Petroleum Hydrocarbons are listed in Appendix A.

1.2. This SOP is applicable to methods 8260B and 8260C, using prep methods 5030A, 5030B, 5030C, 5035, 5035A and Method 624.

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200ºC and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 0.5 to 80 ug/L for waters, 5 to 400 ug/kg for low-level soils, and 50 to 5,000 ug/kg for medium-level soils.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

1.6. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Soil samples may be preserved with sodium bisulfate and purged directly, or preserved by extracting with methanol and diluting into reagent water.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 50°C (50°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. %D (Percent Drift or Percent Difference)

%D is used to refer to both percent drift (Equation 4, Section 12.4.4) and percent difference (Equation 5, Section 12.4.5). The differences between the two calculations, and when they are each applied, are described in Section 12.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Additionally, storage areas for volatile organic test requests are continuously monitored by analysis of a refrigerator holding blank (see WS-QA-0002 for additional information on refrigerator blank analysis).

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered the sample is diluted until foaming is minimized and sample analysis can proceed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Analysts must wear approved cut resistant gloves when opening or closing VOA vials. These can be either Ansell HyFlex NBR, Kevlar, or MAPA Blue-Grip latex. Chemical protective gloves must be worn under or over these gloves, as needed.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of samples or extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.5. Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the**

method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Gas tight micro syringes: 10 uL and larger.

6.2. Gas tight syringe: 5 to 25 mL glass with Luerlock tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.01 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Vials: 40 mL with screw caps and Teflon liners.

6.4.3. Vials: 40 mL with screw caps, Teflon liners, magnetic stir bars. Vials may also contain 5 mL of sodium bisulfate preservative solution.

6.4.4. Volumetric flasks: 10 mL, 50 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipettes: Pasteur.

6.7. pH paper: Wide range.

6.8. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.9. Purge and Trap Device: OI Analytical Eclipse 4660 Purge and Trap Systems. The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.9.2. Trap: Vocarb #10 trap (Tenax / Silica gel / Carbon molecular sieve) is suitable in the analysis. Other traps, such as Vocarb 4000, or Tenax / Silica gel / Charcoal may be used if the Quality Control criteria are met.

6.9.2.1. NOTE: If the Vocarb 4000 trap is used in the analysis catalytic breakdown of certain compounds may occur due to the high desorption temperature required. Thermal decomposition products must be tracked to monitor the trap effectiveness.

6.9.3. Desorber: Capable of rapidly heating the trap to 180ºC

6.9.4. Autosamplers: OI Analytical 4452 A/S; EST Analytical Centurion A/S; Varian Archon A/S: The closed loop autosampler accommodates 40 ml VOC vials with low bleed Teflon septa. For soil analysis, the unit heats the VOC vial to 50ºC and actuates a magnetic stir bar during sample purging in the vial. For water analysis, the unit delivers a measured amount of sample from a sealed VOC vial into the purging cell.

6.10. Gas Chromatograph/Mass Spectrometer System: HP 6890 Gas Chromatograph with HP 5973 mass analyzer or equivalent.

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system is capable of temperature programming.

6.10.2. Gas Chromatographic Columns: DB-624 20m x 0.18 mm ID, 1 µm film thickness or equivalent.

6.10.3. Mass Spectrometer: The mass spectrometer is capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.4. GC/MS interface: Any interface (including direct introduction to the mass

spectrometer) that achieves all acceptance criteria may be used.

6.10.5. Data System:

6.10.5.1. Aquisition Software: Agilent Chemstation Software Version D. The system provides the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.10.5.2. Data Processing Software: Chrom Version 2.1

6.10.5.2.1. The software can search any GC/MS data file for ions of a specific m/e and can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP)

6.10.5.2.2. The software allows integrating the abundances in any EICP between specified times or scan-number limits.

6.10.5.2.3. Spectra can be compared to the NIST Mass Spectral Library. The most recent version is recommended.

7. REAGENTS AND STANDARDS

7.1. Organic Free Water

Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. Organic free water is generated by continuous sparging of deionized or distilled water with helium or nitrogen.

7.2. Gases for GC operation (i.e., carrier): helium, nitrogen

7.3. Methanol -- Purge and Trap Grade

7.4. Calibration Standard

7.4.1. Stock Solutions:

Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -40° to 0°C. Stock standards for non-gases must be replaced at least every 6 months (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Stocks may be replaced sooner if comparison with check standards prepared from an independent source indicates a problem. Stock standards for the gases (bromomethane, chloromethane, chloroethane, vinyl chloride, bromoethane, trichlorofluoromethane, dichlorodifluoromethane) must be replaced every 2 months (from the time of preparation, if prepared in house, or

from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Once opened, gas stock standards are aliquoted to vials, each stored with minimum headspace for up to 2 months. Each vial is used for no more than one week. Stock standards that come with vendor-recommended temperature storage ranges will be stored at these values.

7.4.2. Working standards:

A working solution containing the compounds of interest is prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working surrogate and internal standard solutions that are stored in the autosampler reservoirs are kept at room temperature. However, they are sealed and kept under positive pressure. They are stable for several days under these conditions. Working standards are monitored by comparison to the initial calibration curve. If any of the compounds drift in response from the initial calibration by more than 20% then fresh standards may need to be prepared.

7.4.3. Calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily. These standards are prepared in 50 or 100 mL volumetric flasks according to the recipes in the table section of this SOP.

7.4.4. If stock or secondary dilution standards are purchased in sealed ampules they may be used up to the manufacturers expiration date or within 6 months of opening, whichever is sooner.

7.4.5. Each multipoint calibration curve must be compared against a second source standard that is independent of the solution used for preparing the curve. This practice is to ensure that no adverse drift in response is occurring.

7.5. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 10 for internal standard components.

7.6. Surrogate Standards: Refer to Table 11 for surrogate standard components and spiking levels.

7.7. Laboratory Control Sample and Matrix Spiking Solutions: Refer to Table 12 for LCS/MS/MSD components and spiking levels.

7.8. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration is 25 ng/uL of 4-Bromofluorobenzene in methanol.

7.9. Initial Calibration Verification (ICV): A standard solution containing the analytes present in the calibration standards, but prepared from a source separate from the calibration standards. The ICV should be near or above the mid-point of the calibration range.

7.10. Calibration standards are prepared by injecting a measured volume of the stock standard into a gas tight syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Water samples are normally preserved in the field at pH < 2 with 1:1 hydrochloric acid.

8.1.1. The maximum holding time is 14 days from sampling until the sample is analyzed (samples that are found to be unpreserved are still analyzed within the 14 day holding time per SW-846, however, the lack of preservation should be addressed in the case narrative). If samples are known to be received without preservation, the holding time is 7 days from sampling to analysis based on guidance from method 624. Samples that require the analysis of 2-chloroethylvinylether must be sampled unpreserved. The lab must give prior approval to accepting samples for 2-chloroethylvinylether.

8.1.2. All samples are stored in glass containers with Teflon lined septa at 0° to 6°C, with minimal headspace.

8.2. Solid samples may be received in several ways, which affects the holding time. All solid samples are stored at 0° to 6°C until analysis, unless otherwise noted.

8.2.1. Grab ("bulk") samples: The solid sample used for volatiles analysis is an aliquot taken from the sample container. The container is typically an 8 or 16 ounce jar, or a core tube. These samples have a holding time of 14 days from date of sampling to date of analysis. If a medium-level analysis is required, the holding time does not change.

8.2.2. EnCore samples: The samples are shipped to the laboratory in EnCore sampling sleeves. The laboratory must preserve these within 48 hours of sampling. In general, 3 sleeves are submitted for each sample. Two of these are preserved with sodium bisulfate / H_2O solution, and the third is preserved with methanol, with a holding time of 14 days from sampling to analysis. If effervescence occurs, one of the sodium bisulfate fractions is preserved with water and frozen at -40° to 0°C until analysis. In this case, samples must be analyzed within 14 days of sampling. Alternatively, samples may be extruded into an empty sealed VOA vial and frozen within 48 hours of sampling, then analyzed within 14 days of sampling. Certain programs or states, such as Hawaii, may set a 7 day hold time for frozen samples. Refer to Work Instruction WS-WI-0033 for specific details of handling EnCore samples once received at the laboratory.

8.2.3. Field preserved: Solid samples are taken and preserved in the field with either sodium bisulfate solution, methanol, or frozen in a VOA vial. Samples frozen in encores must be analyzed or transferred to a sealed VOA vial within 48 hours of sampling.

8.3. Methanolic solid extracts may be aliquotted into 2 - 5 mL glass vials or maintained in original 20 mL VOA vials with Teflon lined caps and stored at 0° to 6°C. The extracts are stored with minimal headspace. These extracts must be analyzed within 14 days of collection.

8.4. The maximum holding time for all volatile analyses is 14 days from sample collection.

8.5. At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A. It is no longer included in SW-846 and is likely to generate results that are biased low.

8.6. Holding blanks are prepared, stored, and analyzed in accordance with SOP WS-QA-0002, "Procedure for the Set-Up, Maintenance, and Analysis of Holding Blanks for Volatile Refrigerators".

9. QUALITY CONTROL

9.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this logic, each BFB analysis will normally start a new batch. Batches for laboratory preserved medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together. Batches for field-preserved medium level solids are defined at the initial analysis, and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

9.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, a LCS/LCSD may be used if there is insufficient sample volume for a MS/MSD if batch precision is required by the client or program. A NCM (Non Conformance Memo) must be generated in this case. Refer to the QC Program document (WS-PQA-003) for further details of the batch definition.

9.2. Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

- Corrective action is not required for levels below the reporting limit (i.e., "J" values).

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the QA department and the client.
- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations less than 10 times the method blank are determined in the samples.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers.
- The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low, and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the QA department and the client should take place.
- The DOD QSM criteria for method blanks is no analytes detected greater than ½ the reporting limit (RL). For common laboratory contaminants, it is no analytes detected greater than the RL.

9.2.1. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B" (or other flag as required by the specific client program), and appropriate comments may be made in a narrative to provide further documentation.

9.2.2. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.3. Laboratory Control Samples (LCS)

For each batch of samples, analyze an LCS. The LCS is analyzed after the calibration standard(s), and normally before any samples. The LCS contains all the analytes of interest (see Table 12), and must contain the same analytes as the matrix spike. Upon client request, additional analytes may be added to the LCS. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch. The analyst should monitor the system if non-control analytes are outside of established control limits, and perform corrective action as necessary to prevent re-occurring failures.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that sample surrogate recoveries are good and recoveries are high, and samples were ND for these analytes.)
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.3.1. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.4. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate (MS/MSD). Spiking compounds and levels are given in Table 12. Compare the percent recovery and relative percent difference (RPD) to the historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented using a non-conformance memo.
- If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample volume, then a LCS duplicate may be analyzed if batch precision is required by the client or program. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The MS/MSD must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out, or the spiked analytes will be over the calibration range. Any analytes in the MS/MSD that go over the calibration range due to high analyte levels in the unspiked sample will be anomalized.

9.4.1. Method 624 requires a matrix spike for at least 5% of the samples from each sample site being monitored

9.4.1.1. The spike concentration should be at 20 ug/L or as in compliance monitoring at the regulatory concentration limit.

9.4.1.2. Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria in Table 16.

9.4.2. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.5. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 11. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions of extracted samples):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.

- Re-prepare and reanalyze the sample or flag the data if neither of the above resolves the problem. Table 18 shows the associated target analytes for each surrogate.

It is only necessary to re-prepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that there is a problem with either of the two analyses other than a matrix effect.

9.5.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.5.2. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.6. Internal Standards

Internal standards are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard recoveries are monitored to verify that instrument performance is acceptable. Criteria for calibration standards are delineated in Section 10. If the internal standard data indicate instrument failure, the samples may require reanalysis. If not, the impact on sample data is evaluated and the data is flagged appropriately.

9.6.1. Any samples that do not meet the internal standard criteria for the continuing calibration (Section 10.8.6) must be evaluated for validity. If the change in internal standard response is a matrix effect confined to an individual sample, reanalysis is not necessary. If the change in internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.7. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery ± 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 3 standard deviations.

9.7.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into the LIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.7.2. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.8. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.9. Quality Assurance Summaries

Some clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10. TestAmerica Sacramento QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Summary

Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system. The use of separate calibrations is required for water/medium level soil and low soil matrices.

10.3. Recommended Instrument Conditions

More details of recommended instrument conditions may be found in Table 15.

Gas chromatograph suggested temperature programs	
BFB Analysis	85°C (no hold) 20°C/minute to 160°C (hold 2.0 min) 50°C/minute to 200°C (hold 1.2 min)
Sample Analysis	35°C (hold 2 min) 15°C/minute to 200°C (hold 3.0 min)

10.4. Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 13 for a 50 ng injection of BFB (1 uL of a 50 ng/uL standard). Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB. Method 624 requires a BFB tune at the beginning of each day of analysis.

10.4.1. To evaluate the BFB tune, verify that the software averaged three scans, at the

apex, 1 scan before the apex, and one scan after the apex. Alternatively, the scan at the apex may be evaluated.

10.4.2. Background subtraction shall be employed. The scan used for background subtraction must be before the elution of BFB, but no earlier than 20 scans before the elution of BFB, and may not include part of the peak.

10.5. Initial Calibration

A series of at least five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound (this is referred to as a "five-point calibration" later in this SOP). Typical calibration levels for aqueous samples are: 0.25, 0.5, 1, 4, 20, 40, 60, and 80 µg/L. Some analytes are prepared at higher concentrations due to poor purge performance. Tables 2 and 3 list the calibration levels for each analyte in solid and aqueous samples. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.5.1. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests. For example, the Extended list requires the "normal 8260" standard and the Extended list standards. If acceptable analytical performance can be obtained, the primary and Extended list standards may be analyzed together.

10.5.2. Internal standard calibration is used. The internal standards are listed in Table 10. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the relative response factor (RRF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Equation 1, Section 12, for calculation of relative response factor.

10.5.3. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new (uncalibrated) columns. A new calibration is not required after performing column maintenance, replacing the septum or syringe, or performing other minor maintenance.

10.5.4. With the exception of instances detailed in Policy CA-T-P-002 it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points.

10.6. Evaluating the Initial Calibration

10.6.1. Prior to calculating, ensure that all peak IDs are correct. The compounds listed in Section 12.1.2 may be commonly confused and mis-identified. They may have similar spectra, but typically do not elute near each other. Ensure that these peaks are not assigned the same retention time.

10.6.2. Calculate the average relative response factor for each compound.

10.6.2.1. For method 8260B, verify that the System Performance Check Compounds (SPCC) below meet their minimum average relative response factor.

Method 8260B SPCC Compounds and Minimum Response Factors			
Compound	**Min. RRF**	**Compound**	**Min. RRF**
Chloromethane	0.100	1,1,2,2-Tetrachloroethane	0.300
1,1-Dichloroethane	0.100	Chlorobenzene	0.300
Bromoform	0.100		

If the average RRF for any of the SPCC compounds is less than the minimum specified, the calibration is not valid. The initial calibration must be repeated before analysis may continue. Instrument maintenance may be required.

10.6.2.2. For method 8260C, compare the response factors for each calibration level to the minimums in Table 14. All analytes requiring minimum response factors in the initial calibration must exceed their required value in each level of the calibration curve. Any individual analyte that fails the minimum response factor must have a demonstration of sensitivity (low level CCV at or below the reporting limit) in each analytical batch to demonstrate sensitivity. The analyte must be detected and meet all standard qualitative identification criteria listed in Section 12. This low-level CCV is analyzed following the mid-level CCV (See also Section 10.8).

10.6.3. Calculate the %RSD for each compound.

10.6.4. For method 8260B, evaluate the %RSDs of the Calibration Check Compounds (CCC) listed below, which must be less than 30%.

CCC Compounds	
Vinyl Chloride	1,2-Dichloropropane
1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene

10.6.4.1. If the %RSD for any CCC compound is greater than 30%, the calibration is not valid. The initial calibration must be repeated before analysis may continue. Instrument maintenance may be required.

10.6.4.2. If none of the CCCs are required analytes, project specific calibration specifications may be negotiated with the client.

10.6.5. Evaluate the %RSD of each analyte. For method 8260B, the RSD criteria is less than or equal to 15%. For Method 8260C, the RSD criteria is less than or equal to 20%. For Method 624 the RSD criteria is less than 35%.

10.6.5.1. If the %RSD is within the criteria above for a compound, the average relative response factor is used to quantitate that compound.

10.6.5.2. If the %RSD is greater than the criteria for a compound, attempt a linear curve fit. The linear calibration model may employ weighting such as 1/concentration2 or 1/concentration (see also Section 10.6.7). Method 8260C also permits forcing through zero to improve the fit at the lower end of the calibration curve. No matter what weighting factors or forcing modes are employed, linear curve fits must have a correlation coefficient (r) of ≥ 0.995, or a coefficient of determination (r^2) of ≥ 0.99. In addition, the y intercept should be within +(RL/ISTD) to –1/2(RL/ISTD). If the intercept exceeds this criterion, false positives or negatives could result. If the intercept exceeds +1/2(RL/ISTD), then J values should be evaluated for possible false positives. If the RL is elevated for client or project requirements, use the default RL in the calculation.

Note: The RL value must be in terms of the amount "on-column" on the instrument.

10.6.5.3. If the linear fit does not meet criteria, a second-order ("quadratic curve") fit may be attempted. The second order calibration model may employ weighting such as 1/concentration2 or 1/concentration (see also Section 10.6.7). Method 8260C also permits forcing through zero to improve the fit at the lower end of the calibration curve. No matter what weighting factors or forcing modes are employed, the coefficient of determination (r^2) must be ≥ 0.990 and the y intercept (listed as "b" on the Target ICAL summary page) should be within +(RL/ISTD) to –1/2(RL/ISTD). If the intercept exceeds this criterion, false positives or negatives could result. If the intercept exceeds +1/2(RL/ISTD), then J values should be evaluated for possible false positives. If the RL is elevated for client or project requirements, use the default RL in the calculation

Note: The RL value must be in terms of the amount "on-column" on the instrument.

10.6.6. Whichever calibration model is used (average RRF or a linear or quadratic line fit), it is recommended that the calibration data be quantitated based on the calibration factors, ie, compare the calculated value of each calibration standard for each analyte against the expected amount. In general, the percent different of

these read-backs should not exceed the calibration verification standard acceptance criteria (Section 10.8),

10.6.6.1. Method 8260C indicates that linear calibration models have a greater potential for significant bias at the lower portion of the calibration curve. Consequently, the read-back value for the lowest calibration level should be within ± 30% of the true concentration for each analyte in the standard.

10.6.7. Additional requirements for Method 8260C:

- If more than 10% of the analytes fail both the 20% RSD and the 0.990 correlation coefficient, the initial calibration must be repeated.
- Any individual analyte that fails both the 20% RSD and the 0.990 correlation coefficient must have any positive result flagged as estimated or noted in an NCM. This option is not to be employed on a routine basis.
- Non-detect results for any analyte that fails both 20% RSD and 0.990 correlation coefficient may be reported without flagging if and only if there has been a successful analysis of a CCV at the reporting limit in the same analytical batch. The analyte must be detected and meet all standard qualitative identification criteria listed in Section 12.
- In general, no more than one or two of the poorest performing analytes should fail both 20% RSD and 0.990 correlation coefficient.

10.6.8. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.6.9. Internal Standards

Evaluate the IS response for each calibration level against the IS response in the CCV-level calibration standard. If the internal standard response in a given calibration level is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of the initial calibration performed while the system was malfunctioning is required.

10.6.10. Initial Calibration Verification (ICV) – When available, a second source standard is analyzed with the initial calibration curve. Each compound must meet the %D criteria specified below.

- Method 8260B: Within ± 25%D.

- Method 8260C: Within ± 30%D
- Permitted exceptions to the above criteria are listed below:

Classification	Criteria
Gases:	Up to three gases at ≤30%D (chloroethane, bromomethane, dichlorodifluoromethane)
Ketones:	Up to two ketones at ≤ 30%D (acetone, 2-butanone, 4-methyl-2-pentanone, 2-hexanone)
Poor purgers/Poor Responders:	Carbon disulfide ≤ 40%D 1,4-dioxane ≤ 50%D 2-chloroethylvinyl ether ≤ 35%D Vinyl acetate ≤ 40 %D
Extended list:	≤ 50%D for the following: Iodomethane, Methyl methacrylate Allyl Chloride, Ethyl methacrylate Isopropyl ether, t-1,4-Dichloro-2-butene tert-Butyl ethyl ether, Chloroprene Propionitrile, Acrolein Methacrylonitrile, Acrylonitrile Tetrahydrofuran, Hexane Isobutanol, Cyclohexane tert-Amyl methyl ether

- Under the DOD QSM, the criteria is to be within ± 20%D. The laboratory requests that the following compounds be permitted to meet 30%D when accepting QSM work, if these compounds are included in the requested analyte list:

Acrolein	1,4-Dioxane
Trichlorotrifluoroethane	2-Chloroethyl vinyl ether
t-Butanol	T-1,4-Dichloro-2-Butene
Vinyl Acetate	Allyl Chloride
Isobutanol	

Check QAPP requirements, or ensure that the client is aware of these exceptions.

Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards

10.7. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.8. Continuing Calibration: The initial calibration must be verified every twelve hours. Method 624 requires the initial calibration verification on each working day by the measurement of a QC check sample (20ug/L).

10.8.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 5 calibration standard is generally used as the continuing calibration.

10.8.2. Calculate the RRF for each compound in the standard.

10.8.2.1. For method 8260B, verify that the System Performance Check Compounds (SPCC) below meet their minimum average relative response factor.

Method 8260B SPCC Compounds and Minimum Response Factors			
Compound	**Min. RRF**	**Compound**	**Min. RRF**
Chloromethane	0.100	1,1,2,2-Tetrachloroethane	0.300
1,1-Dichloroethane	0.100	Chlorobenzene	0.300
Bromoform	0.100		

If the average RRF for any of the SPCC compounds is less than the minimum specified, the calibration is not valid. The initial calibration must be repeated before analysis may continue. Instrument maintenance may be required.

10.8.2.2. For method 8260C, compare the response to the minimums in Table 14. If the minimum RRF is not met, the system should be evaluated and corrective action taken prior to sample analysis. Possible issues include degradation of the standard mixture and/or contaminated or active sites within the instrument.

10.8.3. Calculate the %D from the initial calibration for each compound (see Sections 12.4.4 and 12.4.5)

10.8.3.1. For method 8260B, the %D for the CCC compounds must be within ± 20%. The %D for non-CCC compounds must be within ± 40%. If CCCs are not included in the list of compounds requested by the client, all compounds of interested for that client must have the %D within ± 20%.

10.8.3.2. For AFCEE, non-CCC compounds may not exceed ± 25%.

10.8.3.3. For DOD QSM work, the %D for each compounds of concern must be within ± 20%. The laboratory requests that the following compounds be permitted to meet 30%D when accepting QSM work, if these compounds are included in the requested analyte list:

Acrolein	1,4-Dioxane
Trichlorotrifluoroethane	2-Chloroethyl vinyl ether
t-Butanol	T-1,4-Dichloro-2-Butene
Vinyl Acetate	Allyl Chloride
Isobutanol	

10.8.3.4. Method 8260C requires that the %D for each target compound be within ± 20%. Any sample detections for analytes that fail the CCV criteria must be flagged as estimated or described in an NCM. Whenever possible, samples should be reanalyzed with a passing CCV. Samples that are non-detect for analytes that fail the CCV criteria must have a low-level CCV (at or below the reporting limit) in the analytical batch as a sensitivity demonstration. The low-level CCV must meet all criteria for qualitative identification.

10.8.3.5. Method 624 verification of the initial calibration occurs with the analysis of a QC check sample at a concentration of 20 ug/L. For each parameter compare the calculated concentration (Q) with the corresponding calibration acceptance criteria in Table 16.

- If the responses for all paremetrs fall within the acceptable ranges, analysis of samples may begin.
- If any individual Q falls outside the range, repeat the verification for that parameter.
- If the response for that parameter does not fall within the acceptance criteria in the second analysis, a new calibration curve must be analyzed.

10.8.4. The BFB tune and continuing calibration must be acceptable before analysis begins.

10.8.5. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.8.6. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standards, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.8.7. Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample

size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by GC or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample, as described in Section 11.12.

11.3. Sample Analysis

11.3.1. All analytical conditions for samples must be the same as for the initial and continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain an MS/MSD, an LCS, and a method blank.

11.3.2.1. The 12-hour tune period defines the batch. If any re-tuning of the instrument is necessary, or if a period of greater than 12 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation/preservation stage.

11.3.2.2. Laboratory generated QC samples (Method Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.3.2.3. It is not necessary to reanalyze batch QC with reanalysis of samples. However, any reruns must be as part of a valid batch.

11.3.2.4. Before the start of an analytical run, a purge/bake cycle should be run to clean out the system. After this, primers can be run to eliminate active sites and stabilize the system. Although the primer may have the same analytes as a CCV, the primers are NOT processed or evaluated as such. After primers are run the RTC and a cleanout blank (COB) are run. At this point the system should be ready for an analytical run.

11.3.2.5. Regardless of the matrix being evaluated, the analytical sequence starts with an acceptable BFB tuning check, followed by an ICAL of at least five points, or a CCV that meets %D criteria from an existing ICAL, as well as the CCC and SPCC criteria. Analysis continues until 12 hours from the tune analysis has passed.

11.4. Aqueous Sample Analysis

Internal standard and surrogate solutions are added at the rate of 1uL per sample to each sample prior to purging. Refer to Tables 10 and 11 for composition of these solutions.

11.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.4.2. Begin the analytical sequence as described in Section 11.3, above.

11.4.3. Prepare and analyze an LCS (and LCSD if required). Partially fill a 50 mL volumetric flask with reagent water, and spike as noted below. Add reagent water to the line, stopper the flask, and gently invert 3 times to mix. Fill one 40 mL VOA vial from the volumetric flask, and cap it. Place the vial on the instrument. The "normal 8260" LCS is required with all sample analyses; the "AP9" LCS is performed only when requested by the client, or on an annual basis.

Aqueous LCS Spiking			
Solution	Final Volume (mL)	Volume Spiked (uL)	
		Normal 8260 LCS	AP9 LCS
ODD (200 ug/mL)	50	5	--
Megamix (200 ug/mL)	50	5	--
Gasses (200 - 5000 ug/mL)	50	5	--
Vinyl Acetate (200 ug/mL)	50	5	--
MVRADD (200 ug/mL)	50	--	5
Acrolein (5000 ug/mL)	50	--	5

Composition of the solutions above is defined in the Table Section of this SOP.

11.4.4. Follow the LCS with clean-out blanks, as needed.

11.4.5. Prepare and analyze a method blank. Fill a VOA vial with reagent water, and cap. Place the vial on the instrument.

11.4.6. Once LCS and MB have been evaluated, up to 20 field samples may be added to the instrument. If matrix spikes are required, the matrix spike solutions must be added to the vial prior to analysis. Spike samples are decanted into a 50 mL volumetric flask, spiked and decanted into 43mL VOA vials.

Aqueous Sample Spiking in a 50ml Volumetric		
Spiking Standard	Volume Spiked (uL)	
	Normal 8260 MS/SD	AP9 MS/SD
ODD (200 ug/mL)	5	--
Megamix (200 ug/mL)	5	--
Gasses (200 - 5000 ug/mL)	5	--
Vinyl Acetate (200 ug/mL)	5	--
MVRADD (200 ug/mL)	--	5
Acrolein (5000 ug/mL)	--	2

Composition of the solutions above is defined in the Table Section of this SOP.

Note: *Samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation.*

11.4.7. Samples are purged, desorbed and analyzed under the same conditions as standards and QC, according to Table 15.

11.4.8. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.4.9. Check and record the pH of the sample remaining in the VOA vial after analysis is completed.

11.5. Preserving solid samples (in EnCore or ESS Core N' One samplers) for low level analysis (laboratory preservation with sodium bisulfate). (Method 5035 and 5035A) Refer also to the work instruction WS-WI-0033.

Note: The laboratory can accommodate only the 5 g samplers for low-level analysis.

11.5.1. Use pre-prepared VOA vials containing a magnetic stir bar.

11.5.2. Attach a label. The label must not cover the neck of the vial or the autosampler will malfunction.

11.5.3. Tare the scale and weigh the vial (vial, stir bar and label).

11.5.4. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Weigh the vial to the nearest 0.01 g to obtain the weight of soil and record the weight in TALS. Add 5mL of water. Proceed to Section 11.7.

11.5.5. If the sample will not be analyzed immediately, store at -40 to 0 ^{o}C until analysis. Proceed to Section 11.7.

11.6. When preparing solid samples sub-sampled from grab samples for low level analysis (method 5030A), refer to SOP WS-QA-0018 for sub-sampling procedures.

11.6.1. Prepare VOA vials by adding a magnetic stir bar.

11.6.2. Tare the vial, and sub-sample approximately 5 g soil into the vial. Record the mass to the nearest 0.01g.

Note: Less than 5 g soil may be used (to a minimum of 1 g) if slight sample dilution is required. See Section 11.12.

11.6.3. Add 5 mL of reagent water and stir bar to the sample, seal the vial and label.

11.6.4. Proceed immediately to Section 11.7 for analysis.

11.7. Low-Level Solids Analysis

Internal standard and surrogate solutions are added at the rate of 1uL per sample, in 5 mL reagent water to each sample prior to purging. Refer to Tables 10 and 11 for composition of these solutions, and final sample concentrations.

11.7.1. This method is based on purging a heated sediment/soil sample mixed with organic free water containing the surrogate and, if applicable, matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 50°C. Instrument conditions are in table 15.

11.7.2. Begin the analytical sequence as described in Section 11.4, above.

11.7.3. If some of the samples to be analyzed in the batch are preserved with sodium bisulfate are to be analyzed in the batch, use a pre-preserved vial for the method blank. Otherwise, prepare a method blank by adding 5 mL of reagent water to a VOA vial with stir bar. Seal and label the vial.

11.7.4. Prepare an LCS (and LCSD if required) by spiking 5 mL of reagent water with 2.5 uL of each matrix spiking solution. Place in a 40 mL VOA vial with stir-bar, seal and label the vial(s).

Low-level Solid Sample Spiking (5 gram)		
Spiking Standard	Volume Spiked (uL)	
	Normal 8260 MS/SD	AP9 MS/SD
Megamix (100 ug/mL)	2.5	--
ODD (100 ug/mL)	2.5	--
VA (100 ug/mL)	2.5	--
Gasses (100 ug/mL)	2.5	--
Vinyl Acetate (100 ug/mL)	2.5	--
MVRAD (100 ug/mL)	--	2.5
Acrolein (1000 ug/mL)	--	2.5

11.7.5. For matrix spike aliquots, spike as outlined in Section 11.7.4 above.

11.7.6. Soil samples that have low surrogate recovery when analyzed should be reanalyzed once to confirm matrix effect. The optional surrogate Dibromofluoromethane is unstable in basic pH environment and is thus not

recommended to be reported.

11.8. Preparing solid samples field preserved with methanol (without surrogate):

11.8.1. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note in the preservation log.

11.8.2. Proceed to Section 11.9.3, below.

11.9. Preparing solid samples (in EnCore samplers) for medium level analysis (laboratory preservation with methanol). (Methods 5035 and 5035A). Refer also to the work instruction WS-WI-0033.

11.9.1. Tare the scale and record the weight of the VOA vial.

11.9.2. Extrude the (nominal) 5 g sample into the tared vial. Weigh the vial plus soil, and record the sample weight to nearest 0.01g in TALS. Add 5mL of methanol to the VOA vial, seal and store at 0 to 6 ^{o}C.

11.9.3. Spike each sample as noted below. All values are assuming a nominal 5g sample. The addition of spike introduces a slight error (0.4%) into the calculations, which can be neglected.

Methanol Extracted Solid Sample Spiking			
Spiking Standard	Volume Spiked (uL) onto 5 g soil		
	Normal Sample	Normal 8260 LCS/LCSD/MS/SD	AP9 LCS/LCSD/MS/SD
Surrogate (100 ug/mL)	8µL		50
Megamix (100 ug/mL)	--	50µL	--
ODD (100 ug/mL)	--	50µL	
VA (100 ug/mL)	--	50µL	--
Gasses (100 ug/mL)	--	50µL	--
Vinyl Acetate (100 ug/mL)	--	--	50
MVRAD (100 ug/mL)	--	--	50
Acrolein (5000 ug/mL)	--	--	20

11.9.4. Prepare an LCS for each batch by adding the correct amount of matrix spiking and surrogate spiking solutions to 5 mL methanol.

11.9.5. Prepare a method blank for each batch by adding the correct amount of surrogate solution to 5 mL of methanol in a VOA vial.

11.9.6. Shake the samples for two minutes to distribute the methanol throughout the soil.

11.9.7. Best results are obtained by allowing the samples to equilibrate with the methanol for at least 48 hours before continuing with the analysis. Since this is not always possible, be certain to note the preparation time as well as date in the preservation

logbook.

11.9.8. Allow to settle. The extract aliquot is maintained in its original 20 mL VOA vial at 0° to 6°C until analysis. Proceed to Section 11.11 for analysis.

11.10. Methanol Extract Soil Analysis

For field preserved samples, internal standard and surrogate are added by the autosampler prior to purging, as in aqueous analysis (Section 11.4, above). For laboratory preserved samples, surrogate is added at the time of preservation, and the internal standard is added when the methanol solution is added to the VOA vial.

11.10.1. Methanol extracts are analyzed under the same instrumental conditions as aqueous samples, i.e., purging at ambient temperatures.

11.10.2. Begin the analytical sequence as described in Section 11.4, above.

11.10.3. Prepare each sample (including batch QC) by partially filling a 40 mL VOA vial with reagent water. Spike 860 uL methanol extract into the vial. Fill the vial, cap, invert to mix, and place on the instrument.

11.10.3.1. If the sample was field preserved, set the autosampler to add internal standard and surrogate solutions prior to purging.

11.10.3.2. If the sample was laboratory preserved, add 4.3 uL internal standard solution prior to filling and capping the vial.

11.11. Waste Dilution

This is used for waste materials soluble in methanol.

11.11.1. Tare a clean 10 mL volumetric flask. Add approximately 1 g of the waste material and record the mass.

11.11.2. Add about 5-8 mL methanol, and swirl gently to mix. Dilute to the mark with methanol. Prepare sample for analysis as a methanol extract (Section 11.10).

11.11.3. On the instrument, begin the analytical sequence as described in Section 11.4, above. Use the batch QC (method blank and LCS) prepared for aqueous samples (Section 11.4). Recalculate the batch QC as though they were diluted waste, to keep values and units consistent with those of the sample.

11.12. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should range from the concentration of the daily CCV to the high point of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 50% of the upper calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to

bring the largest hit into the concentration range of the daily CCV to the high point of the calibration.

11.12.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

11.12.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request, or in accordance with Federal programs, such as DoD.

11.12.3. Aqueous samples: Prepare the dilution in an appropriate sized volumetric flask (based on the dilution needed) with reagent water. Fill a VOA vial with the dilution and analyze.

11.12.4. If the dilution required would use less than 1 uL of sample, then serial dilutions must be made in volumetric flasks.

11.12.4.1. The diluted concentration should be estimated to be in mid-calibration range.

11.12.5. Low-level solids: if subsampled from grab samples, a smaller amount (down to 1g) may be analyzed. If sample amounts less than 1g are required, or the samples are provided in EnCore samplers, a medium-level (methanol extract) analysis must be performed.

11.12.6. Medium-level (methanol extract) solids: Decrease the volume of methanol spiked into the VOA vial (Section 11.11.3). If the volume of methanol to be spiked is less than 5 uL, prepare a dilution of the methanol first.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.1.2. Anyone evaluating data is trained to know how to handle isomers with similar or identical mass spectra and close elution times. These include:

Mass	**Analytes**
101	Trichlorofluoromethane & Trichlorotrifluoroethane
96	1,1,-Dichloroethene, trans-1,2-Dichloroethene, & cis-1,2-Dichloroethene
43	Vinyl Acetate & 2-Butanone
106, 104	Xylenes (m,p), Xylene (o), and Styrene
105	Isopropylbenzene, 1,3,5-Trimethylbenzene, 1,2,4-Trimethylbenzene, and sec-butylbenzene
146	1,3-Dichlorobenzene, 1,2-Dichlorobenzene, and 1,4-Dichlorobenzene
91	n-propylbenzene, 2-chlorotoluene, 4-chlorotoluene, and n-butylbenzene
180	1,2,4-Trichlorobenzene and 1,2,3-Trichlorobenzene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If a TIC compound is not part of the client target analyte list, but is calibrated on the instrument and is qualitatively and/or quantitatively identifiable, it must be quantitated based on the calibration, and an NCM filed that it was qualitatively and quantitatively reported compared to a known standard that is in control. If the calibration is not in control, then it should still be narrated that it was reported compared to a known standard, however, that standard is not in control.

12.2.2. If the client requests components not associated with the calibration standards, a search of the NIST Library may be made for the purpose of tentative

identification. Guidelines are:

12.2.2.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.2.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.2.4. Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.2.5. Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.2.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.2.2.7. Any compound identified by the library search with a probability of 80% or less will be reported as UNKNOWN only. Any compound identified by the library search with 80% to 100% probability will be reported as the compound selected by the library search program. The only exception will be Aldol Condensate products, which will be reported as “Aldol Condensate Product”, and not by the analyte name.

12.2.2.8. Compounds or peaks present in both the sample and blanks are not reported.

12.3. All manual or re-integration of chromatograms must be documented in accordance with Policy CA-QS-002 and the Sacramento-specific addendum. Documentation includes, as a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration. All manual integrations must undergo a second level review.

12.4. Calculations.

12.4.1. Relative response factor (RRF)

Equation 1

$$RRF = \frac{A_X \times C_{IS}}{A_{IS} \times C_X}$$

Where:

A_X = Area of the characteristic ion for the compound to be measured
A_{IS} = Area of the characteristic ion for the specific internal standard
C_{IS} = Concentration of the specific internal standard, ng/mL
C_X = Concentration of the compound being measured, ng/mL

12.4.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{n} \frac{(X_i - X)^2}{n-1}}$$

Where:

X_i = Value of X at i
N = Number of points
X = Average value of X_i

12.4.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{SD}{\overline{RRF}} \times 100$$

Where:

SD = Standard Deviation (Eq. 2)
$\overline{RRF}$ = Average of the relative response factors

12.4.4. Percent drift – Percent drift is used when average response factor calibration is not used (i.e., for calculating acceptance of continuing calibration of analytes calibrated using a linear or quadratic curve fit). For acceptance of analytes calculated using average response factor, see Section 12.4.5, Percent Difference.

Equation 4

$$\%Drift = \frac{C_{Found} - C_{Expected}}{C_{Expected}} \times 100$$

Where:

C = Concentration

12.4.5. Percent Difference – Percent difference is used when average response factor calibration is used.

Equation 5

$$\%Difference = \frac{RRF_{Std} - \overline{RRF}}{\overline{RRF}} \times 100$$

Where:

$\overline{RRF}$ = Average of the relative response factors

RRF_{std} = Relative Response factor of the continuing calibration verification

12.4.6. Calculating the "on-column" amount in an unknown sample using Average RRF:

Equation 6

$$C_X = \frac{A_X \times C_{IS}}{A_{IS} \times \overline{RRF}}$$

Where:

A_X = Area of the characteristic ion for the compound to be measured
A_{IS} = Area of the characteristic ion for the specific internal standard
C_{IS} = Concentration of the specific internal standard, ng/mL
C_X = Concentration of the compound being measured, ng/mL
$\overline{RRF}$ = Average response factor of the compound.

12.4.6.1. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_X = Area of the total ion chromatogram for the compound to be measured

A_{IS} = Area of the total ion chromatogram for the specific internal standard

$\overline{RRF}$ = Assumed to be equal to 1.

12.4.7. Calculating the "on-column" amount in an unknown sample using a linear fit, not forced through zero. (Based on the "Target" data system).

Equation 7

$$C_X = C_{IS} \times \left(b + \frac{A_X}{A_{IS} \times m1} \right)$$

Where:

A_X = Area of the characteristic ion for the compound to be measured
A_{IS} = Area of the characteristic ion for the specific internal standard
C_{IS} = Concentration of the specific internal standard, ng/mL
C_X = Concentration of the compound being measured, ng/mL
b = Constant as calculated by Target
$m1$ = First degree factor, as calculated by Target

12.4.8. Calculating Sample Concentrations

12.4.8.1. Concentration in an aqueous sample or a low-level solid sample (C_s)

Equation 8

$$C_S = C_X \times \frac{V_D}{V_I}$$

Where:

C_X = Concentration of the compound being measured, ng/mL (Eq. 7)
V_D = Default Purge Aliquot Size (g or mL)
V_I = Actual Purge Aliquot Size (g or mL)

12.4.8.2. Concentration in a medium-level solid sample (C_S)

Equation 9

$$C_S = C_X \times \frac{V_W}{V_I} \times \frac{V_M}{M_S}$$

Where:

C_X = Concentration of the compound being measured, ng/mL (Eq. 7)
V_W = Volume of Water used
V_I = Volume of methanol extract added to water
V_M = Volume of methanol added to solid during preservation
M_S = Mass of solid sample

Note: *Any required percent moisture correction occurs following entry into a LIMS database and not at the instrument. See SOP WS-OP-0013, "Determination of Percent Moisture" for details of determining and reporting soil moisture content.*

12.4.8.3. To calculate the concentration in a medium-level solid sample (C_S) accounting for percent moisture contribution (per Method 8000C), use Equation 9, above, but calculate V_M as noted below:

Equation 10

$$V_M = V_{MP} + \left(\frac{Ms \times \%M}{100} \right)$$

Where:

V_M = Volume of methanol added to solid during preservation plus the water content of the sample
V_{MP} = Volume of methanol added to solid during preservation
M_S = Mass of solid sample
$\%M$ = Percent moisture content of the sample (as measured using SOP WS-OP-0013)

Note: *The calculation above does not replace dry-weight correction. It compensates for the water contribution to the methanol used to preserve/extract the sample only. Any required percent moisture correction occurs following entry into a LIMS database and not at the instrument. See SOP WS-OP-0013, "Determination of Percent Moisture" for details of determining and reporting soil moisture content.*

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. VOA vials containing extracted acidic water and small amounts of methanol. Once analysis is completed, these are collected in plastic lined boxes in the lab. When the box is full, it is transferred to the waste disposal area where the vials are run through the vial eater for disposal.

15.2. VOA vials containing extracted soil samples, which will contain small amounts of methanol and possibly sodium bisulfate. Once analysis is completed, these are collected in plastic lined boxes in the lab. When the box is full, it is transferred to the waste disposal area. The hazardous waste specialist visually inspects each vial with extracted soil. If there is visible liquid in the vial, it is disposed of through the vial eater. If there is no visible liquid, the vial will be placed into a soil collection drum.

15.3. Aqueous acidic waste from sample analysis, which may contain small amounts of methanol. This waste is collected in one gallon plastic carboys. When the carboy is full, it is taken to an H3 waste collection area, and is poured into a blue plastic LLE collection drum. When full to between two and six inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. TestAmerica Sacramento Work Instruction WS-WI-0033, “Preparing Samples Submitted in EnCore Samplers”.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035, 8000B, and 8260B.

16.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 4th Edition, Method 5035A Draft Rev. 1, July 2002.

16.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 2nd Edition, Method 5030A Rev. 1, July 1992.

16.5. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, March 2003, Section 8000C.

16.6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, August 2006, Section 8260C.

16.7. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, May 2003, Section 5030C.

16.8. 40 CFR Appendix A, Part 136, Method 624, March 2014.

17. METHOD MODIFICATIONS

17.1. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

18. ATTACHMENTS

18.1. Table 1 – 8260 Compounds and Reporting Limits

18.2. Table 2 – "Normal 8260" Standard Calibration levels (Aqueous)

18.3. Table 3 – 8260 Mix Liquid Curve Recipe (100 mL final volume)

18.4. Table 4 – Appendix IX Standard Calibration Levels (Aqueous Samples)

18.5. Table 5 – AP9/Oxy Liquid Curve Recipe (50 mL final volume)

18.6. Table 6 – "Normal 8260" Standard Calibration Levels (Solid Analysis)

18.7. Table 7 – "Normal 8260" Standard Low-Level Solid Curve Recipe (5 mL Final Volume)

18.8. Table 8 – Appendix 9 Standard Calibration Levels (Solid Samples)

18.9. Table 9 – Appendix 9 Low-level Solid Curve Recipes (5 mL final volumes)

18.10. Table 10 – Internal Standards

18.11. Table 11 – Surrogate Standards

18.12. Table 12 – LCS and MS/SD Spiking Components and Concentrations

18.13. Table 13 – BFB Key Ion Abundance Criteria

18.14. Table 14 – Characteristic Ions

18.15. Table 15 – Suggested Instrument Conditions

18.16. Table 16 – Calibration and QC Acceptance Criteria – Method 624

18.17. Table 17 – Method Accuracy and Precision as Functions of Concentration – Method 624

18.18. Appendix A – Gas Analysis by GC/MS based on SW-846, CA-LUFT, AK101 and Northwest Methods

19. REVISION HISTORY

19.1. WS-MS-00077, Revision 5.1, Effective 09/05/2014

19.1.1. Section 6.9 – added 'desorber' to list of components at end of sentence.

19.1.2. Section 9.3 – Inserted 'The LCS contains a representative subset of the analytes of interest (see Table 12), and must contain the same analytes as the matrix spike.'

19.1.3. Section 11.4.6 – Inserted 'The LCS contains a representative subset of the analytes of interest (see Table 12), and must contain the same analytes as the matrix spike' and 'Aqueous Sample Spiking in a 100mL Volumetric' table following Section 11.4.6.

19.1.4. Section 11.7.4 - Inserted 'Low Level Solid Spiking' table following Section 11.7.4.

19.1.5. Section 11.9.3 – Inserted 'Methanol Extracted Solid Sample Spiking' table following Section 11.9.3.

19.1.6. Added Section 16.7 - Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, August 2006, Section 5030C.

19.1.7. Table I – Added ethanol to analyte list.

19.1.8. Editorial changes.

19.2. WS-MS-0007, Revision 5.0, Effective 04/15/2014

19.2.1. Added 624 to the SOP title.

19.2.2. Included language in Section 1.1 for the applicability for municipal and industrial discharge.

19.2.3. Added Method 624 to Section 1.2.

19.2.4. Section 9.4.1 had language inserted for the frequency of matrix spike at 5% of the samples from each sample site monitored.

19.2.5. Include BFB frequency at the beginning of each day of analysis in Section 10.4

19.2.6. Method 624 criteria for RSD < 35% inserted in Section 10.6.5.

19.2.7. Sections 10.6.2.2 and 10.8.3.4 expanded to include language from the Corporate Quality Policy Memorandum CA-Q-QM-009.

19.2.8. Inserted Section 10.6.7, additional initial calibration requirements for Method 8260C.

19.2.9. Insert Section 10.8 for the initial calibration verification on each work day and the concentration of the QC check sample.

19.2.10. Inserted Section 10.8.3.5 for language on QC check sample acceptance criteria.

19.2.11. Inserted Table 16 – Calibration and QC Acceptance Criteria – Method 624

19.2.12. Inserted Table 17 – Method Accuracy and Precision as Functions of

Concentration – Method 624

19.2.13. Revision history prior to 2010 has been removed and is available in archived versions of this SOP.

19.3. WS-MS-0007, Revision 4.5, Effective 07/15/2013

19.3.1. Removed reference to Method 8015 (all version) in the Title.

19.3.2. Added references and criteria for Method 8260C to Sections 10.6 and 10.8.

19.3.3. Added commonly confused compounds to Section 12.1.2.

19.3.4. Confirmed the list of excepted compounds for QSM work.

19.3.5. Added references to WS-WI-0033 for preparation of EnCore samples.

19.3.6. Added 8260C minimum RRFs to Table 14.

19.4. WS-MS-0007, Revision 4.4, Effective 02/29/2012

19.4.1. Revised Section 7.5.1 from: "Once opened, gas stock standards are aliquoted to vials, each stored with minimum headspace for up to 2 months. Each vial is used for no more than two weeks" to "Each vial is used for no more than one week."

19.4.2. Revised Section 10.8.3.1 to read: "If the CCCs are not included in the list of compounds for a project, and therefore not included in the calibration standards, than all compounds **must** meet the 20% difference of drift criterion."

19.5. WS-MS-0007, Revision 4.3, Effective 12/23/2011

19.5.1. Inserted Section 8.1 into Appendix I (Gas Analysis by GC/MS Based on SW-846, CALUFT, AK101, and Northwest Methods) outlining the process for ADEC soil sample collection and preservation.

19.5.2. Editorial revisions.

19.6. WS-MS-0007, Revision 4.2, Effective 2/26/2010

19.6.1. Revised Section 6.8.3 to "#10 trap (Tenax/silica gel/carbon molecular sieve)."

19.6.2. Revised Section 8.2.2 to "…are preserved with sodium bisulfate / H_2O solution,".

19.6.3. Revised calibration levels in Tables 1 through 12.

19.6.4. Reversed upper part of Equation 4 and Equation 5 to correct signs in Section 12.4.3 and Section 12.4.4.

Table 1
Sacramento 8260 Compounds and Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		Water µg/L	Low soil µg/kg	Med. Soil µg/kg
Acetone	67-64-1	10	50	500
Acrolein	107-02-8	10	50	500
Acrylonitrile	107-13-1	20	100	500
Benzene	71-43-2	1.0	5.0	50
Bromobenzene	108-86-1	1.0	5.0	50
Bromochloromethane	74-97-5	1.0	5.0	50
Bromodichloromethane	75-27-4	1.0	5.0	50
Bromoform	75-25-2	1.0	5.0	50
Bromomethane	74-83-9	1.0	5.0	50
t-Butanol	75-65-0	50	250	1250
2-Butanone (MEK)	78-93-3	2.0	10	100
n-Butylbenzene	104-51-8	1.0	5.0	50
sec-Butylbenzene	135-98-8	1.0	5.0	50
tert-Butylbenzene	98-06-6	1.0	5.0	50
Carbon disulfide	75-15-0	2.0	10	100
Carbon tetrachloride	56-23-5	1.0	5.0	50
Chlorobenzene	108-90-7	1.0	5.0	50
Chloroprene	126-99-8	2.0	10	100
Dibromochloromethane	124-48-1	1.0	5.0	50
Chloroethane	75-00-3	1.0	5.0	50
2-Chloroethyl vinyl ether	110-75-8	2.0	10	100
Chloroform	67-66-3	1.0	5.0	50
1-Chlorohexane	544-10-5	2.0	10	100
Chloromethane	74-87-3	1.0	5.0	50
Allyl chloride	107-05-1	2.0	10	100
2-Chlorotoluene	95-49-8	1.0	5.0	50
4-Chlorotoluene	106-43-4	1.0	5.0	50
Cyclohexane	110-82-7	2.0	10	100
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	2.0	10	100
1,2-Dibromoethane (EDB)	106-93-4	2.0	10	100
Dibromomethane	74-95-3	1.0	5.0	50
1,2-Dichlorobenzene	95-50-1	1.0	5.0	50
1,3-Dichlorobenzene	541-73-1	1.0	5.0	50
1,4-Dichlorobenzene	106-46-7	1.0	5.0	50
trans-1,4-Dichloro-2-butene	110-57-6	2.0	10	100
Dichlorodifluoromethane (Freon 12)	75-71-8	1.0	5.0	50
1,1-Dichloroethane	75-34-3	1.0	5.0	50
1,2-Dichloroethane	107-06-2	1.0	5.0	50
cis-1,2-Dichloroethene	156-59-2	1.0	5.0	50
trans-1,2-Dichloroethene	156-60-5	1.0	5.0	50
1,1-Dichloroethene	75-35-4	1.0	5.0	50
1,2-Dichloroethene (total)	540-5-90	1.0	5.0	50
1,2-Dichloropropane	78-87-5	1.0	5.0	50

Table 1
Sacramento 8260 Compounds and Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		Water µg/L	Low soil µg/kg	Med. Soil µg/kg
1,3-Dichloropropane	142-28-9	1.0	5.0	50
2,2-Dichloropropane	594-20-7	1.0	5.0	50
cis-1,3-Dichloropropene	10061-01-5	1.0	5.0	50
trans-1,3-Dichloropropene	10061-02-6	1.0	5.0	50
1,1-Dichloropropene	563-58-6	1.0	5.0	50
Diisopropyl ether	108-20-3	2.0	10	100
1,4-Dioxane	123-91-1	50	250	1250
Tert-amyl methyl ether	994-05-8	2.0	10	100
Tert-butyl ethyl ether	637-92-3	2.0	10	100
Ethanol	64-17-5	50	NA	NA
Ethylbenzene	100-41-4	1.0	5.0	50
Ethyl methacrylate	97-63-2	2.0	10.0	100
Hexachlorobutadiene	87-68-3	1.0	5.0	50
Hexane	110-54-3	2.0	10	100
2-Hexanone	591-78-6	2.0	10	100
Iodomethane	74-88-4	2.0	10	100
Isobutyl alcohol	78-83-1	20	100	5000
Isopropylbenzene	98-82-8	1.0	5.0	50
Isopropyl ether	108-20-3	2.0	10	100
p-Isopropyltoluene	99-87-6	1.0	5.0	50
Methacrylonitrile	126-98-7	2.0	10	100
Methylene chloride	75-09-2	1.0	10	100
Methyl methacrylate	80-62-6	2.0	10	100
4-Methyl-2-pentanone (MIBK)	108-10-1	2.0	10	100
Methyl tert-butyl ether (MTBE)	1634-04-4	2.0	10	100
Naphthalene	91-20-3	1.0	5.0	50
Propionitrile	107-12-0	2.0	10	100
n-Propylbenzene	103-65-1	1.0	5.0	50
Styrene	100-42-5	1.0	5.0	50
1,1,1,2-Tetrachloroethane	630-20-6	1.0	5.0	50
1,1,2,2-Tetrachloroethane	79-34-5	1.0	5.0	50
Tetrachloroethene	127-18-4	1.0	5.0	50
Tetrahydrofuran	109-99-9	5.0	NA	NA
Toluene	108-88-3	1.0	5.0	50
1,2,3-Trichlorobenzene	87-61-6	1.0	5.0	50
1,2,4-Trichlorobenzene	120-82-1	1.0	5.0	50
1,1,1-Trichloroethane	71-55-6	1.0	5.0	50
1,1,2-Trichloroethane	79-00-5	1.0	5.0	50
Trichloroethene	79-01-6	1.0	5.0	50
Trichlorofluoromethane (Freon 11)	75-69-4	1.0	5.0	50
1,2,3-Trichloropropane	96-18-4	1.0	5.0	50
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	2.0	10	100
1,2,4-Trimethylbenzene	95-63-6	1.0	5.0	50

Table 1
Sacramento 8260 Compounds and Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		Water µg/L	Low soil µg/kg	Med. Soil µg/kg
1,3,5-Trimethylbenzene	108-67-8	1.0	5.0	50
Vinyl acetate	108-05-4	2.0	10	100
Vinyl chloride	75-01-4	1.0	5.0	50
m-Xylene & p-Xylene	136777-61-2	1.0	5.0	50
o-Xylene	95-47-6	1.0	5.0	50
Xylenes (total)	1330-20-7	1.0	5.0	50

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be recovered from acid preserved samples, including solids preserved with sodium bisulfate solution.

Table 2

"Normal 8260" Standard Calibration Levels (Aqueous and Medium Level Solid Analysis)

Compound	Stock ug/mL	Calibration Level ug/L							
		1	2	3	4	5	6	7	8
Surrogate									
Dichlorofluoromethane (Surrogate-optional)	100	--	2.0	4.0	10	20	40	60	80
1,2-Dichloroethane-d4 (Surrogate)	100	--	2.0	4.0	10	20	40	60	80
Toluene-d8 (Surrogate)	100	--	2.0	4.0	10	20	40	60	80
4-Bromofluorobenzene (Surrogate)	100	--	2.0	4.0	10	20	40	60	80
TCL Mix									
Dichlorodifluoromethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Chloromethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Vinyl chloride	200	0.25	0.5	1.0	4.0	20	40	60	80
Chloroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Bromomethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Trichlorofluoromethane	200	0.25	0.5	1.0	4.0	20	40	60	80
1,1-Dichloroethene	200	0.25	0.5	1.0	4.0	20	40	60	80
Carbon disulfide	200	0.25	0.5	1.0	4.0	20	40	60	80
Methylene chloride	200	0.25	0.5	1.0	4.0	20	40	60	80
trans-1,2-Dichloroethene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,1-Dichloroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
2,2-Dichloropropane	200	0.25	0.5	1.0	4.0	20	40	60	80
cis-1,2-Dichloroethene	200	0.25	0.5	1.0	4.0	20	40	60	80
Bromochloromethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Chloroform	200	0.25	0.5	1.0	4.0	20	40	60	80
1,1,1-Trichloroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
1,1-Dichloropropene	200	0.25	0.5	1.0	4.0	20	40	60	80
Carbon tetrachloride	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2-Dichloroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Benzene	200	0.25	0.5	1.0	4.0	20	40	60	80
Trichloroethene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2-Dichloropropane	200	0.25	0.5	1.0	4.0	20	40	60	80
Dibromomethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Bromodichloromethane	200	0.25	0.5	1.0	4.0	20	40	60	80
cis-1,3-Dichloropropene	200	0.25	0.5	1.0	4.0	20	40	60	80
Toluene	200	0.25	0.5	1.0	4.0	20	40	60	80
trans-1,3-Dichloropropene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,1,2-Trichloroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Tetrachloroethene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,3-Dichloropropane	200	0.25	0.5	1.0	4.0	20	40	60	80
Dibromochloromethane	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2-Dibromoethane	200	0.25	0.5	1.0	4.0	20	40	60	80
1-Chlorohexane	200	0.25	0.5	1.0	4.0	20	40	60	80
Ethylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
Chlorobenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,1,1,2-Tetrachloroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
m and p Xylenes	400	0.50	1.0	2.0	8.0	40	80	120	160
o-xylene	200	0.25	0.5	1.0	4.0	20	40	60	80
Styrene	200	0.25	0.5	1.0	4.0	20	40	60	80
Bromoform	200	0.25	0.5	1.0	4.0	20	40	60	80

Table 2									
"Normal 8260" Standard Calibration Levels (Aqueous and Medium Level Solid Analysis)									
Compound	Stock ug/mL	Calibration Level ug/L							
		1	2	3	4	5	6	7	8
Isopropylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2,3-Trichloropropane	200	0.25	0.5	1.0	4.0	20	40	60	80
Bromobenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,1,2,2-Tetrachloroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
n-Propylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
2-Chlorotoluene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,3,5-Trimethylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
4-Chlorotoluene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2,4-Trimethylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
T-Butylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
Sec-Butylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,3-Dichlorobenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
p-Isopropyltoluene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,4-Dichlorobenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2-Dichlorobenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
n-Butylbenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2-Dibromo-3-chloropropane	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2,3-Trichlorobenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
Hexachlorobutadiene	200	0.25	0.5	1.0	4.0	20	40	60	80
Naphthalene	200	0.25	0.5	1.0	4.0	20	40	60	80
1,2,3-Trichlorobenzene	200	0.25	0.5	1.0	4.0	20	40	60	80
ODD Mix									
Acetone	200	0.25	0.5	1.0	4.0	20	40	60	80
Acrylonitrile	2000	2.5	5.0	10	40	200	400	600	800
Trichlorotrifluoroethane	200	0.25	0.5	1.0	4.0	20	40	60	80
Methyl *tert*-butyl ether (MTBE)	200	0.25	0.5	1.0	4.0	20	40	60	80
2-Butanone	200	0.25	0.5	1.0	4.0	20	40	60	80
1,4-Dioxane	5000	6.25	12.5	25	100	500	1000	1500	2000
t-Butanol	5000	6.25	12.5	25	100	500	1000	1500	2000
2-Chloroethyl vinyl ether	200	0.25	0.5	1.0	4.0	20l	40	60	80
4-Methyl-2-pentanone	200	0.25	0.5	1.0	4.0	20	40	60	80
2-Hexanone	200	0.25	0.5	1.0	4.0	20	40	60	80
Vinyl Acetate									
Vinyl acetate	200	0.25	0.5	1.0	4.0	20	40	60	80
Ethanol									
Ethanol	100,000	50	200	1000	2000	3000	4000	8000	12000
Methanolic TVPH Surrogate									
a,a,a-Trifluorotoluene	100	--	2.0	4.0	10	20	40	60	80

Table 3

8260 Liquid Curve
100 mL

Calibration Point	Final Volume (mL)	Concentration (µg/L)	Auto IS (500 µg/mL)	Surrogate (100 µg/mL)	*ICAL mix (5-10 µg/mL)	TCLA & TCLB (200 µg/mL)	ODD #1 & #2 (200 – 5000 µg/mL)	Vinyl Acetate (2000 µg/mL)
1	100	0.25/0.50	1.0 µL	--	5.0 µL	---	---	---
2	100	0.5/1.0	1.0 µL	1 µL	10 µL	---	---	---
3	100	1.0/2.0	1.0 µL	2 µL	20 µL	---	---	---
4	100	4	1.0 µL	4 µL	---	2 µL	2 µL	2 µL
5 (CCV)	100	20	1.0 µL	20 µL	---	10 µL	10 µL	10 µL
6	100	40	1.0 µL	40 µL	---	20 µL	20 µL	20 µL
7	100	60	1.0 µL	60 µL	---	30 µL	30 µL	30 µL
8	100	80	1.0 µL	80 µL	---	40 µL	40 µL	40 µL

BFB	1µL	500µg/mL	Direct Inject

*ICAL Mix (5 – 10µg/mL = 400µL FV (350µL MeOH + 10µL TCLA + 10µL TCLB + 10µL ODD #1 + 10µL ODD #2 + 10µL VA)

Table 4								
Appendix IX Standard Calibration Levels (Aqueous and Medium Level Solid Analysis)								
Compound	Stock Solution ug/mL	Calibration Level (ug/L)						
		Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
AP9 A solution								
Iodomethane	200	2	4	10	20	40	60	80
Allyl Chloride	200	2	4	10	20	40	60	80
Isopropyl ether	200	2	4	10	20	40	60	80
tert-Butyl ethyl ether	200	2	4	10	20	40	60	80
Propionitrile	200	2	4	10	20	40	60	80
Methacrylonitrile	200	2	4	10	20	40	60	80
Tetrahydrofuran	200	2	4	10	20	40	60	80
Isobutanol	2000	20	40	100	200	400	600	800
tert-Amyl methyl ether	200	2	4	10	20	40	60	80
Methyl methacrylate	200	2	4	10	20	40	60	80
Ethyl methacrylate	200	2	4	10	20	40	60	80
t-1,4-Dichloro-2-butene	200	2	4	10	20	40	60	80
AP9 B								
Hexane	200	2	4	10	20	40	60	80
Cyclohexane	200	2	4	10	20	40	60	80
Chloroprene	200	2	4	10	20	40	60	80
ACROLEIN								
Acrolein	2000	20	40	100	200	400	600	800

Table 5				
AP9/Oxy Curve Recipe (100mL final Volume) (Aqueous and Medium Level Solid Analysis)				
Calib Level	IS (100 ug/mL)	AP9 A (200-5000) ug/mL	AP9 B 200 ug/mL	Acrolein 2000 ug/mL
2	10 uL	1 uL	1 uL	1 uL
3	10 uL	2 uL	2 uL	2 uL
4	10 uL	5 uL	5 uL	5 uL
5	10 uL	10 uL	10 uL	10 uL
6	10 uL	20 uL	20 uL	20 uL
7	10 uL	30 uL	30 uL	30 uL
8	10 uL	40 uL	40 uL	40 uL

Table 6									
"Normal 8260" Calibration Level (Low Level Solid Analysis)									
Compound	Stock ug/mL	Calibration Level ug/L							
		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Surrogate									
Dichlofluoromethane (Surrogate, Optional)	100	--	--	10	20	50	100	200	400
1,2-Dichloroethane-d4 (Surrogate)	100	--	--	10	20	50	100	200	400
Toluene-d8 (Surrogate)	100	--	--	10	20	50	100	200	400
4-Bromofluorobenzene (Surrogate)	100	--	--	10	20	50	100	200	400
TCL Mix									
Dichlorodifluoromethane	200	2.5	5	10	20	50	100	200	400
Chloromethane	200	2.5	5	10	20	50	100	200	400
Vinyl chloride	200	2.5	5	10	20	50	100	200	400
Chloroethane	200	2.5	5	10	20	50	100	200	400
Bromomethane	200	2.5	5	10	20	50	100	200	400
Trichlorofluoromethane	200	2.5	5	10	20	50	100	200	400
1,1-Dichloroethene	200	2.5	5	10	20	50	100	200	400
Carbon disulfide	200	2.5	5	10	20	50	100	200	400
Methylene chloride	200	2.5	5	10	20	50	100	200	400
trans-1,2-Dichloroethene	200	2.5	5	10	20	50	100	200	400
1,1-Dichloroethane	200	2.5	5	10	20	50	100	200	400
2,2-Dichloropropane	200	2.5	5	10	20	50	100	200	400
cis-1,2-Dichloroethene	200	2.5	5	10	20	50	100	200	400
Bromochloromethane	200	2.5	5	10	20	50	100	200	400
Chloroform	200	2.5	5	10	20	50	100	200	400
1,1,1-Trichloroethane	200	2.5	5	10	20	50	100	200	400
1,1-Dichloropropene	200	2.5	5	10	20	50	100	200	400
Carbon tetrachloride	200	2.5	5	10	20	50	100	200	400
1,2-Dichloroethane	200	2.5	5	10	20	50	100	200	400
Benzene	200	2.5	5	10	20	50	100	200	400
Trichloroethene	200	2.5	5	10	20	50	100	200	400
1,2-Dichloropropane	200	2.5	5	10	20	50	100	200	400
Dibromomethane	200	2.5	5	10	20	50	100	200	400
Bromodichloromethane	200	2.5	5	10	20	50	100	200	400
cis-1,3-Dichloropropene	200	2.5	5	10	20	50	100	200	400
Toluene	200	2.5	5	10	20	50	100	200	400
trans-1,3-Dichloropro-pene	200	2.5	5	10	20	50	100	200	400
1,1,2-Trichloroethane	200	2.5	5	10	20	50	100	200	400

Table 6									
"Normal 8260" Calibration Level (Low Level Solid Analysis)									
Compound	Stock ug/mL	**Calibration Level ug/L**							
		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Tetrachloroethene	200	2.5	5	10	20	50	100	200	400
1,3-Dichloropropane	200	2.5	5	10	20	50	100	200	400
Dibromochloromethane	200	2.5	5	10	20	50	100	200	400
1,2-Dibromomethane	200	2.5	5	10	20	50	100	200	400
1-Chlorohexane	200	2.5	5	10	20	50	100	200	400
Ethylbenzene	200	2.5	5	10	20	50	100	200	400
Chlorobenzene	200	2.5	5	10	20	50	100	200	400
1,1,1,2-Tetrachloroethane	200	2.5	5	10	20	50	100	200	400
m and p Xylenes	200	2.5	5	10	20	50	100	200	400
o-xylene	200	2.5	5	10	20	50	100	200	400
Styrene	200	2.5	5	10	20	50	100	200	400
Bromoform	200	2.5	5	10	20	50	100	200	400
Isopropylbenzene	200	2.5	5	10	20	50	100	200	400
1,2,3-Trichloropropane	200	2.5	5	10	20	50	100	200	400
Bromobenzene	200	2.5	5	10	20	50	100	200	400
1,1,2,2-Tetrachloroethane	200	2.5	5	10	20	50	100	200	400
n-Propylbenzene	200	2.5	5	10	20	50	100	200	400
2-Chlorotoluene	200	2.5	5	10	20	50	100	200	400
1,3,5-Trimethylbenzene	200	2.5	5	10	20	50	100	200	400
4-Chlorotoluene	200	2.5	5	10	20	50	100	200	400
1,2,4-Trimethylbenzene	200	2.5	5	10	20	50	100	200	400
t-Butylbenzene	200	2.5	5	10	20	50	100	200	400
Sec-Butylbenzene	200	2.5	5	10	20	50	100	200	400
1,3-Dichlorobenzene	200	2.5	5	10	20	50	100	200	400
p-Isopropyltoluene	200	2.5	5	10	20	50	100	200	400
1,4-Dichlorobenzene	200	2.5	5	10	20	50	100	200	400
1,2-Dichlorobenzene	200	2.5	5	10	20	50	100	200	400
n-Butylbenzene	200	2.5	5	10	20	50	100	200	400
1,2-Dibromo-3-chloropropane	200	2.5	5	10	20	50	100	200	400
1,2,3-Trichlorobenzene	200	2.5	5	10	20	50	100	200	400
Hexachlorobutadiene	200	2.5	5	10	20	50	100	200	400
Naphthalene	200	2.5	5	10	20	50	100	200	400
1,2,3-Trichlorobenzene	200	2.5	5	10	20	50	100	200	400
ODD Mix									
Acetone	200	2.5	5	10	20	50	100	200	400
Acrylonitrile	2000	25	50	100	200	500	1000	2000	4000
Trichlorotrifluoroethane	200	2.5	5	10	20	50	100	200	400

Table 6									
Extended List Standard Calibration Levels (Low Level Solid Analysis)									
Compound	**Stock ug/mL**	**Calibration Level ug/mL**							
		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Methyl *tert*-butyl ether (MTBE)	200	2.5	5	10	20	50	100	200	400
2-Butanone	200	2.5	5	10	20	50	100	200	400
1,4-Dioxane	5000	62.5	125	250	500	1250	2500	5000	10000
t-Butanol	5000	62.5	125	250	500	1250	2500	5000	10000
2-Chloroethyl vinyl ether	200	2.5	5	10	20	50	100	200	400
4-Methyl-2-pentanone	200	2.5	5	10	20	50	100	200	400
2--Hexanone	200	2.5	5	10	20	50	100	200	400
Vinyl Acetate									
Vinyl acetate	200	2.5	5	10	20	50	100	200	400

Table 7

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8260 Low Soil Curve

5.0g in (10ml H2O)

Calibration point	Concentration (ug/Kg)	Final Volume	Internal Standard (250 ug/mL) Autospike	Surrogate (250 ug/mL)	TCL A (100 ug/mL)	TCL B (100 ug/mL)	ODD Mix #1 (100 ug/mL)	ODD Mix #2 (100 ug/mL)	Vinyl Acetate (100 ug/mL)
1	2.5	10 mL	1.0 uL	2.5	N/A	N/A	N/A	N/A	N/A
2	5	10 mL	1.0 uL	5 uL (a)	N/A	N/A	N/A	N/A	N/A
3	10	10 mL	1.0 uL	10 uL (a)	N/A	N/A	N/A	N/A	N/A
4	20	10 mL	1.0 uL	20 uL (a)	N/A	N/A	N/A	N/A	N/A
5 (CCV)	50	10 mL	1.0 uL	1 uL	2.5 uL	2.5 uL	2.5 uL	2.5 uL	2.5 uL
6	100	10 mL	1.0 uL	2 uL	5 uL	5 uL	5 uL	5 uL	5 uL
7	200	10 mL	1.0 uL	4 uL	10 uL	10 uL	10 uL	10 uL	10 uL
8	400	10 mL	1.0 uL	8 uL	20 uL	20 uL	20 uL	20 uL	20 uL

(a) = 20uL of Surrogate, 50ul of TCL A, TCL B, Odd Mix #1, Odd Mix #2, and Vinyl Acetate diluted to FV of 1.0mL in MeOH.

Analyst: _____ Date: _____

Daily QC

Calibration point	Final Volume	Concentration (ug/L)	Internal Standard (250 ug/mL) Autospike	Surrogate (500 ug/mL) Autospike	Secondary Sources (100 ug/mL)
LCS/LCSD	10 mL	50	1.0 uL	1.0 uL	2.5 uL
MS/MSD	10 mL	50	1.0 uL	1.0 uL	2.5 uL
Sample	10 mL	50	1.0 uL	1.0 uL	N/A

Analyst: _____ Date: _____

Comments : _____

Q:\FORMS\Checklists\QA-633_8260 Low Soil Curve QA-633 04/29/2012 RE

Table 8									
Extended List Standard Calibration Levels (Low-Level Solid Samples)									
Compound	Stock Solution ug/mL	Calibration Level (ng/g)							
		Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
AP9/OXY solution									
Iodomethane	200	2.5	5	10	20	50	100	200	400
Allyl Chloride	200	2.5	5	10	20	50	100	200	400
Isopropyl ether	200	2.5	5	10	20	50	100	200	400
tert-Butyl ethyl ether	200	2.5	5	10	20	50	100	200	400
Propionitrile	200	2.5	5	10	20	50	100	200	400
Methacrylonitrile	200	2.5	5	10	20	50	100	200	400
Tetrahydrofuran	200	2.5	5	10	20	50	100	200	400
Isobutanol	2000	25	50	100	200	500	1000	2000	4000
tert-Amyl methyl ether	200	2.5	5	10	20	50	100	200	400
Methyl methacrylate	200	2.5	5	10	20	50	100	200	400
Ethyl methacrylate	200	2.5	5	10	20	50	100	200	400
t-1,4-Dichloro-2-butene	200	2.5	5	10	20	50	100	200	400
CHLOROPRENE									
Chloroprene	200	2.5	5	10	20	50	100	200	400
ACROS MIX									
Acrolein	2000	25	50	100	200	500	1000	2000	4000
Acrylonitrile	2000	25	50	100	200	500	1000	2000	4000
HEXANE/CYCLOHEXANE									
Hexane	200	2.5	5	10	20	50	100	200	400
Cyclohexane	200	2.5	5	10	20	50	100	200	400

Table 9						
Extended list Low-Level Solid Curve Recipe (5.0 g 10 mL purge)						
Calib Level	Concentration	Final Volume H2O	IS (250 µg/mL) Autospike	AP9 A (200-5000 µg,/mL)	AP9 B (200-5000 µg,/mL)	Acrolein (2000 µg/mL)
1	2.5	10 mL	1.0 uL	2.5 µL (a)		2.5 µL (b)
2	5	10 mL	1.0 uL	5 µL (a)		5 µL (b)
3	10	10 mL	1.0 uL	10 µL (a)		10 µL (b)
4	20	10 mL	1.0 uL	20 µL (a)		20 µL (b)
5(ccv)	50	10 mL	1.0 uL	50 µL (a)		50 µL (b)
6	100	10 mL	1.0 uL	2.5 µL	2.5 µL	2.5 µL
7	200	10 mL	1.0 uL	5 µL	5 µL	5 µL
8	400	10 mL	1.0 uL	10 µL	10 µL	10 µL

(a) = 250µL of the A P9 A and AP9 B combined and diluted to 1.0 mL in MeOH.
(b) = 250 µL of the Acrolein standard diluted to 1.0 mL in water.l

Table 10				
Internal Standards				
Internal Standard Component	Quantitation ion	Solution Concentration (µg/mL)	Concentration in Sample	
			Aqueous (ng/mL)	Soil (ng/g)
Aqueous and Medium Solids				
Dibromofluoromethane (optional)	111	50	10	500
1,4-Difluorobenzene	114	50	10	500
Chlorobenzene-d5	117	50	10	500
1,4-Dichlorobenzene-d4	152	50	10	500
Low-Level Solids				
1,4-Difluorobenzene	114	500	--	50
Chlorobenzene-d5	117	500	--	50
1,4-Dichlorobenzene-d4	152	500	--	50

Table 11			
Surrogate Standards			
Surrogate Standard Component	Solution Concentration (µg/mL)	Concentration in Sample	
		Aqueous (ng/mL)	Soil (ng/g)
Aqueous and Medium Solids			
Dibromofluoromethane (optional)	100	20	1000
1,2-Dichloroethane-d_4	100	20	1000
Toluene-d_8	100	20	1000
4-Bromofluorobenzene	100	20	1000
Low-Level Solids			
Dibromofluoromethane	500	--	100
1,2-Dichloroethane-d_4	500	--	100
Toluene-d_8	500	--	100
4-Bromofluorobenzene	500	--	100

Table 12
LCS and MS/SD Spiking Components and Concentrations

Compound	Solution Concentration (ug/ml)	Spiking Level		
		Water (ug/L)	Low Soil (ug/kg)	Medium Soil (ug/kg)
Acetone	200	20	100	1000
Acetonitrile	200	20	100	1000
Acrolein	2000	200	1000	10,000
Acrylonitrile	2000	200	1000	10,000
Benzene (1)	200	20	100	1000
Bromobenzene	200	20	100	1000
Bromochloromethane	200	20	100	1000
Bromodichloromethane	200	20	100	1000
Bromoform	200	20	100	1000
Bromomethane	200	20	100	1000
t-Butanol	5050	500	2500	25,000
2-Butanone (MEK)	200	20	100	1000
n-Butylbenzene	200	20	100	1000
sec-Butylbenzene	200	20	100	1000
tert-Butylbenzene	200	20	100	1000
Carbon disulfide	200	20	100	1000
Carbon tetrachloride	200	20	100	1000
Chlorobenzene (1)	200	20	100	1000
Chloroprene	200	20	100	1000
Dibromochloromethane	200	20	100	1000
Chloroethane	200	20	100	1000
2-Chloroethyl vinyl ether	200	20	100	1000
Chloroform	200	20	100	1000
1-Chlorohexane	200	20	100	1000
Chloromethane	200	20	100	1000
Allyl chloride	200	20	100	1000
2-Chlorotoluene	200	20	100	1000
4-Chlorotoluene	200	20	100	1000
1,2-Dibromo-3-chloropropane (DBCP)	200	20	100	1000
1,2-Dibromoethane (EDB)	200	20	100	1000
Dibromomethane	200	20	100	1000
1,2-Dichlorobenzene	200	20	100	1000
1,3-Dichlorobenzene	200	20	100	1000
1,4-Dichlorobenzene	200	20	100	1000
trans-1,4-Dichloro-2-butene	200	20	100	1000
Dichlorodifluoromethane (Freon 12)	200	20	100	1000
1,1-Dichloroethane	200	20	100	1000
1,2-Dichloroethane	200	20	100	1000
cis-1,2-Dichloroethene	200	20	100	1000
trans-1,2-Dichloroethene	200	20	100	1000
1,1-Dichloroethene (1)	200	20	100	1000

Table 12
LCS and MS/SD Spiking Components and Concentrations

Compound	Solution Concentration (ug/ml)	Spiking Level		
		Water (ug/L)	Low Soil (ug/kg)	Medium Soil (ug/kg)
1,2-Dichloropropane	200	20	100	1000
1,3-Dichloropropane	200	20	100	1000
2,2-Dichloropropane	200	20	100	1000
cis-1,3-Dichloropropene	200	20	100	1000
trans-1,3-Dichloropropene	200	20	100	1000
1,1-Dichloropropene	200	20	100	1000
1,4-Dioxane	5000	500	2500	25,000
Tert-amyl methyl ether	200	20	100	1000
Tert-butyl ethyl ether	200	20	100	1000
Ethanol	100,000	2000	NA	NA
Ethylbenzene	200	20	100	1000
Ethyl methacrylate	200	20	100	1000
Hexachlorobutadiene	200	20	100	1000
Hexane	200	20	100	1000
2-Hexanone	200	20	100	1000
Iodomethane	200	20	100	1000
Isobutyl alcohol	2000	500	2500	25,000
Isopropylbenzene	200	20	100	1000
Isopropyl ether	200	20	100	1000
p-Isopropyltoluene	200	20	100	1000
Methacrylonitrile	200	20	100	1000
Methylene chloride	200	20	100	1000
Methyl methacrylate	200	20	100	1000
4-Methyl-2-pentanone (MIBK)	200	20	100	1000
Methyl tert-butyl ether (MTBE)	200	20	100	1000
Naphthalene	200	20	100	1000
Propionitrile	200	20	100	1000
n-Propylbenzene	200	20	100	1000
Styrene	200	20	100	1000
1,1,1,2-Tetrachloroethane	200	20	100	1000
1,1,2,2-Tetrachloroethane	200	20	100	1000
Tetrachloroethene	200	20	100	1000
Toluene (1)	200	20	100	1000
1,2,3-Trichlorobenzene	200	20	100	1000
1,2,4-Trichlorobenzene	200	20	100	1000
1,1,1-Trichloroethane	200	20	100	1000
1,1,2-Trichloroethane	200	20	100	1000
Trichloroethene (1)	200	20	100	1000
Trichlorofluoromethane (Freon 11)	200	20	100	1000
1,2,3-Trichloropropane	200	20	100	1000
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	200	20	100	1000

Table 12
LCS and MS/SD Spiking Components and Concentrations

Compound	Solution Concentration (ug/ml)	Spiking Level		
		Water (ug/L)	Low Soil (ug/kg)	Medium Soil (ug/kg)
1,2,4-Trimethylbenzene	200	20	100	1000
1,3,5-Trimethylbenzene	200	20	100	1000
Vinyl acetate	200	20	100	1000
Vinyl chloride	200	20	100	1000
m-Xylene & p-Xylene	200	40	200	2000
o-Xylene	200	20	100	1000

(1) Laboratory default control analytes.

Table 13
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 14					
Characteristic Ions & Minimum Response Factors					
Compound	**Approx Retention Time (min)**	**Primary[4]**	**Second-ary**	**Tertiary**	**Min RRF (8260C)**
Internal Standards					
1,4-Difluorobenzene	7.467	114	63	88	--
Chlorobenzene-d5	12.395	117			--
1,4 Dichlorobenzene-d4	16.908	152			--
Surrogates					
Dibromofluoromethane (optional)	4.281	111	113		--
1,2-Dichloroethane-d4 [1]	6.777	65	102	67	--
Toluene-d8	9.852	98	100	99	--
4-Bromofluorobenzene	14.642	95	174	176	--
Target Analytes					
Dichlorodifluoromethane (Freon 12)	1.81	85	87	50	0.100
Chloromethane	1.988	50	52	49	0.100
Vinyl chloride	2.086	62	64		0.100
Bromomethane	2.411	94	96	93	0.100
Chloroethane	2.5	64	66	49	0.100
Trichlorofluoromethane (Freon 11) [1,2]	2.776	101	151	103	0.100
Acrolein [1,2]	3.229	56	55		0.01
1,1-Dichloroethene [2]	3.338	96	61	98	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) 3	3.357	101	151	103	0.100
Acetone [1,2]	3.397	43	58		0.100
Iodomethane	3.495	142	127		0.01
Carbon disulfide	3.584	76	78		0.100
Acetonitrile [1,2]	3.751	40	41	39	NA
Allyl chloride	3.761	76	41	39	0.1
Methylene chloride	3.909	84	49	86	0.100
t-Butanol [3]	4.125	59	41		0.01
Acrylonitrile	4.214	53	52		0.01
trans-1,2-Dichloroethene	4.264	96	61	98	0.100
Methyl tert-butyl ether (MTBE) [3]	4.264	73	41	57	0.100
1,1-Dichloroethane	4.796	63	65	83	0.200
Vinyl acetate	4.905	43	86	44	0.01
Chloroprene [3]	4.924	53	88		0.01

Table 14					
Characteristic Ions & Minimum Response Factors					
Compound	**Approx Retention Time (min)**	**Primary[4]**	**Second-ary**	**Tertiary**	**Min RRF (8260C)**
Isopropylbenzene [1,2]	4.933	45	87	43	0.100
Tert-butyl ethyl ether [3]	4.943	59	87		0.01
2,2-Dichloropropane	5.595	77	41	97	0.100
cis-1,2-Dichloroethene	5.605	96	61	98	0.100
2-Butanone (MEK) [1,2,5]	5.634	43	72	57	0.100
Propionitrile	5.762	54	55		0.050
Methacrylonitrile	5.929	41	39	67	0.050
Bromochloromethane	5.93	128	49	130	0.100
Tetrahydrofuran [3]	6.066	42	41	72	0.010
Chloroform	6.068	83	85	47	0.200
1,1,1-Trichloroethane	6.324	97	61	99	0.100
1,1-Dichloropropene [2]	6.561	75	39	110	0.050
Carbon tetrachloride	6.57	117	119	121	0.100
Isobutyl alcohol	6.865	43	74		0.010
Benzene	6.866	78	77		0.500
1,2-Dichloroethane	6.896	62	64	98	0.100
Tert-amyl methyl ether [3]	7.091	73	45		0.050
Trichloroethene	7.871	95	130	132	0.200
1,2-Dichloropropane [2]	8.207	63	62	64	0.100
Dibromomethane	8.394	93	174	172	0.050
Methyl methacrylate [1,2]	8.462	41	69		0.050
1,4-Dioxane	8.482	88	58		0.001
Bromodichloromethane [2]	8.67	83	85	47	0.200
2-Chloroethyl vinyl ether [2]	9.202	63	106	43	0.050
cis-1,3-Dichloropropene [2]	9.419	75	77	110	0.200
4-Methyl-2-pentanone (MIBK) [1,2]	9.705	43	58	100	0.100
Toluene	9.961	92	91	65	0.400
trans-1,3-Dichloropropene [2]	10.375	75	77	110	0.100
Ethyl methacrylate	10.611	69	41		0.050
1,1,2-Trichloroethane [1,2]	10.68	97	83	99	0.100
Tetrachloroethene [1,2]	10.917	166	164	129	0.200
1,3-Dichloropropane	10.976	76	78		0.100
2-Hexanone	11.203	43	58	100	0.010
Dibromochloromethane	11.36	129	127	208	0.100
1,2-Dibromoethane (EDB)	11.538	107	109		0.100
Chlorobenzene	12.454	112	77	114	0.500

Table 14					
Characteristic Ions & Minimum Response Factors					
Compound	**Approx Retention Time (min)**	**Primary[4]**	**Second-ary**	**Tertiary**	**Min RRF (8260C)**
1-Chlorohexane [3]	12.484	91	41	55	0.100
1,1,1,2-Tetrachloroethane [2]	12.622	131	133	117	0.100
Ethylbenzene [1,2]	12.691	106	91	51	0.100
m-Xylene & p-Xylene	12.908	91	106	77	0.100
o-Xylene	13.647	91	106		0.300
Styrene	13.676	104	103	78	0.300
Bromoform [2]	14.002	173	171	175	0.100
Isopropyl ether [3]	14.386	105	120	77	0.050
Bromobenzene [2]	14.899	156	77	58	0.100
1,1,2,2-Tetrachloroethane [2]	14.987	83	85	95	0.300
1,2,3-Trichloropropane [2]	15.027	75	110	112	0.100
trans-1,4-Dichloro-2-butene [1,2]	15.105	89	75	53	0.100
n-Propylbenzene	15.184	91	120		0.100
2-Chlorotoluene	15.303	91	126	63	0.100
4-Chlorotoluene	15.529	91	126	63	0.100
1,3,5-Trimethylbenzene	15.559	105	120		0.100
tert-Butylbenzene	16.17	119	91		0.100
1,2,4-Trimethylbenzene	16.278	105	120		0.100
sec-Butylbenzene	16.623	105	134	91	0.100
1,3-Dichlorobenzene	16.781	146	148	111	0.600
p-Isopropyltoluene	16.939	119	91	134	0.100
1,4-Dichlorobenzene	16.958	146	148	111	0.500
1,2-Dichlorobenzene	17.688	146	148	111	0.400
n-Butylbenzene	17.757	91	92	134	0.100
1,2-Dibromo-3-chloropropane (DBCP)	19.255	75	157	155	0.050
1,2,4-Trichlorobenzene [2]	20.516	180	182	184	0.200
Hexachlorobutadiene	20.773	225	227	223	0.100
Naphthalene	20.812	128	51		0.100
1,2,3-Trichlorobenzene [2]	21.108	180	182	184	0.100
Hexane Ethanol	3.222 2.45	57 45	56 46		0.100

[1] Primary ion differs from Method 8260B, Table 5.

[2] Confirmation ions differ from Method 8260B, Table 5.

[3] Characteristic ions for this analyte are not listed in Method 8260B, Table 5.

[4] The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

[5] m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

Table 15	
Suggested Instrumental Conditions	
Electron Energy	70 volts (nominal)
Mass Range	35-350 amu
Scan Time	To give at least 5 scans/peak but not to exceed 2 sec/scan
Initial Column Temperature/Hold Time	35°C for 2.0 minutes
Column Temperature Program	35 - 200°C at 15°C/min.
Injector Temperature	220°C
Transfer Line Temperature	150°C
Source Temperature	200°C
GC Carrier Gas	Helium at 37 cm/sec
Purge Gas/Flow	Helium at 40 ml/min
Purge Time/Temperature	11 min at 50°C (Soils) 11 min at 50°C (Waters)
Purging Temperature	50°C
Trap Desorb Time/Temperature	0.5 min at 190 °C
Trap Bake Time/Temperature	7 min at 210 °C

Table 16—Calibration and QC Acceptance Criteria—Method 624[a]

Parameter	Range for Q (µg/L)	Limit for s (µg/L)	Range for X (µg/L)	Range for P, P_s (%)
Benzene	12.8−27.2	6.9	15.2−26.0	37−151
Bromodichloromethane	13.1−26.9	6.4	10.1−28.0	35−155
Bromoform	14.2−25.8	5.4	11.4−31.1	45−169
Bromomethane	2.8−37.2	17.9	D−41.2	D−242
Carbon tetrachloride	14.6−25.4	5.2	17.2−23.5	70−140
Chlorobenzene	13.2−26.8	6.3	16.4−27.4	37−160
Chloroethane	7.6−32.4	11.4	8.4−40.4	14−230
2-Chloroethylvinyl ether	D−44.8	25.9	D−50.4	D−305
Chloroform	13.5−26.5	6.1	13.7−24.2	51−138
Chloromethane	D−40.8	19.8	D−45.9	D−273
Dibromochloromethane	13.5−26.5	6.1	13.8−26.6	53−149
1,2-Dichlorobenzene	12.6−27.4	7.1	11.8−34.7	18−190
1,3-Dichlorobenzene	14.6−25.4	5.5	17.0−28.8	59−156
1,4-Dichlorobenzene	12.6−27.4	7.1	11.8−34.7	18−190
1,1-Dichloroethane	14.5−25.5	5.1	14.2−28.5	59−155
1,2-Dichloroethane	13.6−26.4	6.0	14.3−27.4	49−155
1,1-Dichlorothene	10.1−29.9	9.1	3.7−42.3	D−234
trans-1,2-Dichloroethene	13.9−26.1	5.7	13.6−28.5	54−156
1,2-Dichloropropane	6.8−33.2	13.8	3.8−36.2	D−210
cis-1,3-Dichloropropene	4.8−35.2	15.8	1.0−39.0	D−227
trans-1,3-Dichloropropene	10.0−30.0	10.4	7.6−32.4	17−183
Ethyl benzene	11.8−28.2	7.5	17.4−26.7	37−162
Methylene chloride	12.1−27.9	7.4	D−41.0	D−221
1,1,2,2-Tetrachloroethane	12.1−27.9	7.4	13.5−27.2	46−157
Tetrachloroethene	14.7−25.3	5.0	17.0−26.6	64−148
Toluene	14.9−25.1	4.8	16.6−26.7	47−150
1,1,1-Trichloroethane	15.0−25.0	4.6	13.7−30.1	52−162
1,1,2-Trichloroethane	14.2−25.8	5.5	14.3−27.1	52−150
Trichloroethene	13.3−26.7	6.6	18.6−27.6	71−157
Trichlorofluoromethane	9.6−30.4	10.0	8.9−31.5	17−181
Vinyl chloride	0.8−39.2	20.0	D−43.5	D−251

Q= Concentration measured in QC check sample, in µg/L (Section 10.8.3.5).
s= Standard deviation of four recovery measurements, in µg/L (Section 13).
X= Average recovery of four recovery measurements, in µg/L (Section 13).
P, P_s= Percent recovery measured.
D= Detected; result must be greater than zero.
[a]Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

Table 17—Method Accuracy and Precision as Functions of Concentration—Method 624

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
Benzene	0.93C+2.00	0.26X−1.74	0.25X−1.33
Bromodichloromethane	1.03C−1.58	0.15X+0.59	0.20X+1.13
Bromoform	1.18C−2.35	0.12X+0.36	0.17X+1.38
Bromomethane[a]	1.00C	0.43X	0.58X
Carbon tetrachloride	1.10C−1.68	0.12X+0.25	0.11X+0.37
Chlorobenzene	0.98C+2.28	0.16X−0.09	0.26X−1.92
Chloroethane	1.18C+0.81	0.14X+2.78	0.29X+1.75
2-Chloroethylvinyl ether[a]	1.00C	0.62X	0.84X
Chloroform	0.93C+0.33	0.16X+0.22	0.18X+0.16
Chloromethane	1.03C+0.81	0.37X+2.14	0.58X+0.43
Dibromochloromethane	1.01C−0.03	0.17X−0.18	0.17X+0.49
1,2-Dichlorobenzene[b]	0.94C+4.47	0.22X−1.45	0.30X−1.20
1,3-Dichlorobenzene	1.06C+1.68	0.14X−0.48	0.18X−0.82
1,4-Dichlorobenzene[b]	0.94C+4.47	0.22X−1.45	0.30X−1.20
1,1-Dichloroethane	1.05C+0.36	0.13X−0.05	0.16X+0.47
1,2-Dichloroethane	1.02C+0.45	0.17X−0.32	0.21X−0.38
1,1-Dichloroethene	1.12C+0.61	0.17X+1.06	0.43X−0.22
trans-1,2,-Dichloroethene	1.05C+0.03	0.14X+0.09	0.19X+0.17
1,2-Dichloropropane[a]	1.00C	0.33X	0.45X
cis-1,3-Dichloropropene[a]	1.00C	0.38X	0.52X
trans-1,3-Dichloropropene[a]	1.00C	0.25X	0.34X
Ethyl benzene	0.98C+2.48	0.14X+1.00	0.26X−1.72
Methylene chloride	0.87C+1.88	0.15X+1.07	0.32X+4.00
1,1,2,2-Tetrachloroethane	0.93C+1.76	0.16X+0.69	0.20X+0.41
Tetrachloroethene	1.06C+0.60	0.13X−0.18	0.16X−0.45
Toluene	0.98C+2.03	0.15X−0.71	0.22X−1.71
1,1,1-Trichloroethane	1.06C+0.73	0.12X−0.15	0.21X−0.39
1,1,2-Trichloroethane	0.95C+1.71	0.14X+0.02	0.18X+0.00
Trichloroethene	1.04C+2.27	0.13X+0.36	0.12X+0.59
Trichloroflouromethane	0.99C+0.39	0.33X−1.48	0.34X−0.39
Vinyl chloride	1.00C	0.48X	0.65X

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
S^r=Expected single analyst standard deviation of measurements at an average concentration found of X, in µg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of X, in µg/L.
C=True value for the concentration, in µg/L.
X=Average recovery found for measurements of samples containing a concentration of C, in µg/L.
[a]Estimates based upon the performance in a single laboratory.[13]
[b]Due to chromatographic resolution problems, performance statements for these

Table 18 – Surrogate associations of the target analytes

Analytes	CAS #	Surrogate	IS	Analyte Type (1)
1,1,2-Trichloroethane	79-00-5	1,2-Dichloroethane	1,4-Diflourobenzene	A
1,1-Dichloropropene	563-58-6	1,2-Dichloroethane	1,4-Diflourobenzene	A
1,2-Dichloroethane	107-06-2	1,2-Dichloroethane	1,4-Diflourobenzene	A
1,2-Dichloropropane	78-87-5	1,2-Dichloroethane	1,4-Diflourobenzene	A
1,4-Dioxane	123-91-1	1,2-Dichloroethane	1,4-Diflourobenzene	A
2-Chloroethyl vinyl ether	110-75-8	1,2-Dichloroethane	1,4-Diflourobenzene	A
4-Methyl-2-pentanone (MIBK)	108-10-1	1,2-Dichloroethane	1,4-Diflourobenzene	A
Benzene	71-43-2	1,2-Dichloroethane	1,4-Diflourobenzene	A
Carbon tetrachloride	56-23-5	1,2-Dichloroethane	1,4-Diflourobenzene	A
cis-1,3-Dichloropropene	10061-01-5	1,2-Dichloroethane	1,4-Diflourobenzene	A
Dibromomethane	74-95-3	1,2-Dichloroethane	1,4-Diflourobenzene	A
Dichlorobromomethane	75-27-4	1,2-Dichloroethane	1,4-Diflourobenzene	A
Ethyl methacrylate	97-63-2	1,2-Dichloroethane	1,4-Diflourobenzene	A
Methyl methacrylate	80-62-6	1,2-Dichloroethane	1,4-Diflourobenzene	A
Toluene	108-88-3	1,2-Dichloroethane	1,4-Diflourobenzene	A
trans-1,3-Dichloropropene	10061-02-6	1,2-Dichloroethane	1,4-Diflourobenzene	A
Trichloroethene	79-01-6	1,2-Dichloroethane	1,4-Diflourobenzene	A
1,1,2,2-Tetrachloroethane	79-34-5	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,2,3-Trichlorobenzene	87-61-6	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,2,3-Trichloropropane	96-18-4	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,2,4-Trichlorobenzene	120-82-1	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,2,4-Trimethylbenzene	95-63-6	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,2-Dibromo-3-Chloropropane	96-12-8	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,2-Dichlorobenzene	95-50-1	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,3,5-Trimethylbenzene	108-67-8	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,3-Dichlorobenzene	541-73-1	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,4-Dichlorobenzene	106-46-7	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
2-Chlorotoluene	95-49-8	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
4-Chlorotoluene	106-43-4	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
4-Isopropyltoluene	99-87-6	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
Bromobenzene	108-86-1	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
Hexachlorobutadiene	87-68-3	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
Isopropylbenzene	98-82-8	4-Bromofluorobenzene	Chlorobenzene-d5	A
Naphthalene	91-20-3	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A

Analytes	CAS #	Surrogate	IS	Analyte Type (1)
n-Butylbenzene	104-51-8	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
N-Propylbenzene	103-65-1	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
sec-Butylbenzene	135-98-8	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
tert-Butylbenzene	98-06-6	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
trans-1,4-Dichloro-2-butene	110-57-6	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4	A
1,1,1-Trichloroethane	71-55-6	Dibromofluoromethane	1,4-Diflourobenzene	A
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Dibromofluoromethane	1,4-Diflourobenzene	A
1,1-Dichloroethane	75-34-3	Dibromofluoromethane	1,4-Diflourobenzene	A
1,1-Dichloroethene	75-35-4	Dibromofluoromethane	1,4-Diflourobenzene	A
2,2-Dichloropropane	594-20-7	Dibromofluoromethane	1,4-Diflourobenzene	A
2-Butanone (MEK)	78-93-3	Dibromofluoromethane	1,4-Diflourobenzene	A
2-Chloro-1,3-butadiene (Chloroprene)	126-99-8	Dibromofluoromethane	1,4-Diflourobenzene	A
2-Methyl-2-propanol (t-butanol)	75-65-0	Dibromofluoromethane	1,4-Diflourobenzene	A
3-Chloro-1-propene	107-05-1	Dibromofluoromethane	1,4-Diflourobenzene	A
Acetone	67-64-1	Dibromofluoromethane	1,4-Diflourobenzene	A
Acrolein	107-02-8	Dibromofluoromethane	1,4-Diflourobenzene	A
Acrylonitrile	107-13-1	Dibromofluoromethane	1,4-Diflourobenzene	A
Bromomethane	74-83-9	Dibromofluoromethane	1,4-Diflourobenzene	A
Butadiene	106-99-0	Dibromofluoromethane	1,4-Diflourobenzene	A
Carbon disulfide	75-15-0	Dibromofluoromethane	1,4-Diflourobenzene	A
Chlorobromomethane	74-97-5	Dibromofluoromethane	1,4-Diflourobenzene	A
Chloroethane	75-00-3	Dibromofluoromethane	1,4-Diflourobenzene	A
Chloroform	67-66-3	Dibromofluoromethane	1,4-Diflourobenzene	A
Chloromethane	74-87-3	Dibromofluoromethane	1,4-Diflourobenzene	A
cis-1,2-Dichloroethene	156-59-2	Dibromofluoromethane	1,4-Diflourobenzene	A
Cyclohexane	110-82-7	Dibromofluoromethane	1,4-Diflourobenzene	A
Dichlorodifluoromethane	75-71-8	Dibromofluoromethane	1,4-Diflourobenzene	A
Ethanol	64-17-5	Dibromofluoromethane		A
Ethyl bromide	74-96-4	Dibromofluoromethane	1,4-Diflourobenzene	A
Hexane	110-54-3	Dibromofluoromethane	1,4-Diflourobenzene	A
Iodomethane	74-88-4	Dibromofluoromethane	1,4-Diflourobenzene	A
Isobutyl alcohol	78-83-1	Dibromofluoromethane	1,4-Diflourobenzene	A
Isopropyl alcohol	67-63-0	Dibromofluoromethane	1,4-Diflourobenzene	A
Isopropyl ether	108-20-3	Dibromofluoromethane	1,4-Diflourobenzene	A

Analytes	CAS #	Surrogate	IS	Analyte Type (1)
Methacrylonitrile	126-98-7	Dibromofluoromethane	1,4-Diflourobenzene	A
Methyl tert-butyl ether	1634-04-4	Dibromofluoromethane	1,4-Diflourobenzene	A
Methylene Chloride	75-09-2	Dibromofluoromethane	1,4-Diflourobenzene	A
n-Heptane	142-82-5	Dibromofluoromethane	1,4-Diflourobenzene	A
Propionitrile	107-12-0	Dibromofluoromethane	1,4-Diflourobenzene	A
Tert-amyl methyl ether	994-05-8	Dibromofluoromethane	1,4-Diflourobenzene	A
Tert-butyl ethyl ether	637-92-3	Dibromofluoromethane	1,4-Diflourobenzene	A
Tetrahydrofuran	109-99-9	Dibromofluoromethane	1,4-Diflourobenzene	A
trans-1,2-Dichloroethene	156-60-5	Dibromofluoromethane	1,4-Diflourobenzene	A
Trichlorofluoromethane	75-69-4	Dibromofluoromethane	1,4-Diflourobenzene	A
Vinyl acetate	108-05-4	Dibromofluoromethane	1,4-Diflourobenzene	A
Vinyl chloride	75-01-4	Dibromofluoromethane	1,4-Diflourobenzene	A
1,2-Dichloroethane-d4 (Surr)	17060-07-0	NA	1,4-Diflourobenzene	S
1,4-Dichlorobenzene-d4	3855-82-1	NA	NA	IS
1,4-Difluorobenzene	540-36-3	NA	NA	IS
4-Bromofluorobenzene (Surr)	460-00-4	NA	Chlorobenzene-d5	S
Chlorobenzene-d5	3114-55-4	NA	NA	IS
Dibromofluoromethane (Surr)	1868-53-7	NA	1,4-Diflourobenzene	S
n-Nonane	111-84-2	NA	NA	A
n-Octane	111-65-9	NA	NA	A
Toluene-d8 (Surr)	2037-26-5	NA	1,4-Diflourobenzene	S
Trifluorotoluene (Surr)	98-08-8	NA	NA	S
1,1,1,2-Tetrachloroethane	630-20-6	Toluene-d8	Chlorobenzene-d5	A
1,3-Dichloropropane	142-28-9	Toluene-d8	Chlorobenzene-d5	A
1-Chlorohexane	544-10-5	Toluene-d8	Chlorobenzene-d5	A
2-Hexanone	591-78-6	Toluene-d8	Chlorobenzene-d5	A
Bromoform	75-25-2	Toluene-d8	Chlorobenzene-d5	A
Chlorobenzene	108-90-7	Toluene-d8	Chlorobenzene-d5	A
Chlorodibromomethane	124-48-1	Toluene-d8	Chlorobenzene-d5	A
Ethylbenzene	100-41-4	Toluene-d8	Chlorobenzene-d5	A
Ethylene Dibromide	106-93-4	Toluene-d8	Chlorobenzene-d5	A
m-Xylene & p-Xylene	179601-23-1	Toluene-d8	Chlorobenzene-d5	A
o-Xylene	95-47-6	Toluene-d8	Chlorobenzene-d5	A
Styrene	100-42-5	Toluene-d8	Chlorobenzene-d5	A
Tetrachloroethene	127-18-4	Toluene-d8	Chlorobenzene-d5	A

1 A =Analyte/Parameter, IS = Internal Standard, S = Surrogate

1. SCOPE AND APPLICATTION

1.1. This method describes sample preparation and extraction for the analysis of volatile petroleum hydrocarbons. The procedures used for the petroleum hydrocarbons are based on SW-846, California LUFT, and ADEC methodology. Refer to Table A1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are soluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soil samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

2. SUMMARY OF METHOD

Refer to Section 2 of the main body of this SOP for a summary of the method.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of the method.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for Safety Information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for equipment and supplies.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the main body of this SOP for reagents and standards as well. The information below is specific to hydrocarbon analysis only.

7.2. Neat Standard Materials

Occasionally, hydrocarbon fuels are not available as stock solutions from commercial vendors. In these instances, neat materials may be acquired directly from the client.

Such neat materials expire following a period of 3 years from the date of receipt, or sooner if problems such as pattern degradation or linearity loss occur.

7.3. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for petroleum hydrocarbons are stored at -40 to -0°C. Other stock standard solutions are stored at 0 to 6ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Stock standards must be replaced at least every 6 months (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Stocks may be replaced sooner if comparison with check standards prepared from an independent source indicates a problem.

7.4. Calibration standards are prepared by injecting a measured volume of the stock standard into a gas tight syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

7.5. Gasoline Standards

Due to the differences between fuels available in California (RFG, or Reformulated Gasoline) and those in the rest of the United States, it is recommended that a commercially available composite gasoline standard be used. (Example: XHc Unleaded gasoline composite standard, #30205 from Restek Corp).

8. SAMPLE PRESERVATION AND STORAGE

See Section 8 of the main body of this SOP for information regarding sample preservation and storage. Some state requirements, e.g., ADEC may require that only field methanol-preserved samples are acceptable.

8.1. If samples are to be analyzed for ADEC, the following preservation is required.

8.1.1. Soil or sediment samples must be collected in appropriately sized containers and submerged in surrogated methanol.

8.1.2. Solid samples must be colleted into tared jars with a Teflon-lined septum fused to the lid. Jars should be 4 oz. or larger if appropriate. 25 mL aliquots of methanol (including 1.2 mL of a surrogate solution of α,α,α,-trifluorotoluene at 50 ug/mL, or equivalent) should be added to the soil until the sample is submerged.

8.1.3. It is extremely important that the weight of the jar, the weight of the methanol/surrogate solution, and the weight of the sample collected be known. These must either be measured directly, or sufficient information documented

so that these weights can be calculated.

8.1.4. Although it is not necessary to refrigerate all methanol preserved samples at 0 to 6°C after collection and until analysis is complete, the collected samples must be kept below 25°C.

8.1.5. The maximum holding time for soil and sediment samples collected under this Section is 28 days.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the main body of this SOP for general quality control requirements.

9.2. For Method AK 101 prepare and analyze an LCS Duplicate sample as well as an LCS sample for each component of interest (GRO, BTEX) for each analytical batch.

9.3. The surrogate compound for pre-preserved medium level (methanol) soils is a,a,a-triflourotoluene. When this compound is used, the instrument is calibrated with it as well, so that recoveries can be accurately calculated.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for additional calibration information. Note that surrogates are calculated using Method 8260 and specific ion fragments.

10.2. External calibration is used. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given Tables A7 and A8.

10.3. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC/MS using the technique that will be used for samples. The ratio of the peak response to the mass or concentration injected may be used to prepare a calibration curve. Peak response is generally measured as the area of the peak.

Equation A 1

$$Calibration\ Factor\ (CF) = \frac{Area\ of\ Peak(s)}{Concentration\ of\ Standard\ (ng/ml\ or\ ug/ml)}$$

10.4. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the % RSD of the response factor or

calibration factor of each analyte is ≤ 20%. If an analyte exceeds the criteria, then a regression line (linear or curved) may be attempted.

10.4.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the **normal** concentration/response behavior of the detector.

10.4.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is ≤ 20%. The equation for average response factor is:

Equation A2

$$\textit{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.4.3. Linear regression

The linear fit uses the following function:

Equation A3

$$x = \frac{(y-b)}{a}$$

Where: y = Instrument response

x = Concentration

a = Slope

b = Intercept

10.4.4. Quadratic curve

The quadratic curve uses the following function:

Equation A4

$$x = \frac{-b + \sqrt{b^2 - 4a(c-y)}}{2a}$$

Where: x = concentration

y = instrument response

a = 2nd order coefficient (curvature)

b = 1st order coefficient (slope)

c = constant (intercept)

10.5. Evaluation of calibration curves

The %RSD from the calibration curve is used to evaluate the initial calibration. If a regression line is used, the coefficient of determination (r2) shall be greater than or equal to 0.990, or the correlation coefficient (r) shall be greater than or equal to 0.995, whichever is appropriate to the regression fit used.

10.6. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response should be less than ± ½ the reporting limit for the analyte.
- The RSD for average response factors for each component must be ≤ 20%.
- When RSD is inappropriate, either the correlation coefficient (r) must be ≥ 0.995, or the coefficient of determination (r^2) must be ≥ 0.990.

10.7. Initial Calibration (ICAL)

10.7.1. Analyze an ICV with the calibration curve. This verification standard is used as a quality control check to verify the accuracy of the calibration.

10.7.2. Low level soil samples must be purged at 50ºC; therefore the calibration curve must also be purged at 50ºC.

10.7.3. A separate calibration, purged at ambient temperature, must be used for aqueous samples. The aqueous calibration may be used for methanolic soil extracts.

10.7.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.7.5. At the beginning of each initial calibration curve for gasoline or other hydrocarbon fuel mixtures, a carbon range standard is run to determine start and end points for the peak summing range for the mixture.

10.7.5.1. See Table A10 for details of this carbon range standard.

10.7.5.2. See Section 10.13 for setting the peak summing range.

10.7.5.3. As this mixture is used for setting retention times based on the total ion chromatogram, it may be analyzed prior to the BFB required before sample analysis.

10.7.6. Quantitating hydrocarbon mixtures:

Starting and ending retention times for quantitation are determined for each fuel. The peak areas between the starting and ending times are summed and used to generate a response factor. This factor is used to quantitate sample results, and depending on the client requirements, this factor may be applied to the same retention range as the standard, or to a different range.

10.8. Integration

10.8.1. When evaluating the initial calibration, also evaluate hydrocarbon pattern integration.

10.8.1.1. The integrated peaks should run from the point in the chromatogram prior to peaks to a region following the mixture's peaks in a fairly straight line.

10.8.2. The default integration parameters generated as a result of evaluating the initial calibration should remain in effect until the initial calibration is reanalyzed.

10.9. Initial Calibration Verification (ICV) -- When available, a second source standard is analyzed with the initial calibration curve. Each compound of the second source calibration must be within ± 15% of its expected value, when evaluated with respect to the calibration curve. Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards

10.10. Continuing Calibration Verification (CCV)

10.10.1. Refer to Table A9 for the preparation of CCV standard(s).

10.10.2. The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, and at the end of every 12 hour analysis sequence. The mid-level calibration standard used at the start of the analysis must be a different concentration than the mid-level calibration standard used for subsequent calibration verifications. Recommended concentrations are noted in Table A9. Because this standard is evaluated using the total ion chromatogram, rather than specific ion fragments, it may be analyzed after the 12 hours following the BFB tune has expired.

10.10.3. The petroleum hydrocarbon elution profile is compared to the initial calibration to verify that shifting has not occurred since initial calibration. If an alkane range standard has been analyzed, it is checked to verify that

shifting has not occurred since the retention time windows for the petroleum hydrocarbon were set.

10.10.4. Any individual compound with %D ≤ 15% meets the calibration criteria. For Method AK101 the CCV can have a %D ≤25%.

10.10.5. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration and second source standard.

10.10.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample, etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria, then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV, then the preceding samples have not been successfully bracketed but analysis may continue.

10.10.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed, then the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples.

10.10.8. If highly contaminated samples are expected, it is acceptable to analyze blanks or primers at any point in the run.

10.10.9. % Difference is calculated as follows:

Equation A5

$$\%\ Difference = \frac{|\ Expected\ value - Calculated\ value\ |}{Expected\ value} \times 100\%$$

10.10.10. Corrective Actions for Continuing Calibration

If the %D for each analyte of concern is greater than 15%, corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the %D still varies by more than + 15%, a new calibration curve must be prepared.

10.10.11. Corrective Action for Samples

Any samples injected after the last good continuing calibration standard must be reinjected, unless one of the following conditions is satisfied:

10.10.11.1. If the CCV response is elevated and the preceding samples are non-detect for all analytes of concern, an NCM may be filed and the data reported.

10.10.11.2. If the samples have already been analyzed twice, and it is apparent that matrix effects are causing the out of control event, an NCM may be filed and the data reported.

10.11. Non-standard analytes are sometimes requested. In the case of these fuels, it may be acceptable to analyze a single standard for pattern identification daily, rather than a five point initial calibration, provided that the QA department and the client agree with this strategy.

10.11.1. Positives which match the pattern of the non-standard analyte may be quantitated based on a standard reference if the elution range is similar and the client agrees to this. In this instance, "unknown hydrocarbons" are quantitated based on the default material for the fraction (e.g., diesel, motor oil, or gasoline, based on elution range).

10.11.2. Otherwise, a five-point initial calibration must be run and samples with positives must be reanalyzed for quantitation. If the analyte is not detected, the non-detect may be reported and no further action is necessary.

10.12. Daily Retention Time Windows

The retention time windows must be updated at the beginning of each analytical sequence by the injection of a retention time window mix.

11. PROCEDURE

11.1. Refer to Section 11 of the main body of this SOP for information regarding sample preparation and other procedures. Procedures specific to hydrocarbon analysis are presented below.

11.2. Analytical Sequence

11.2.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 15 of the main body of this SOP for a 50 ng injection of BFB (1 uL of a 50ng/uL standard). Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

11.2.2. After the BFB, the analytical sequence starts with an ICAL of at least five points, or a CCV that meets %D criteria from an existing ICAL.

11.2.3. A CCV must be analyzed at least once every 12 hours when samples are being

analyzed, and at the end of the sequence. If there is a break in the analytical sequence of greater than 12 hours, then a new CCV (and BFB) run must be analyzed before proceeding with the sequence.

11.2.4. A CCV consists of mid-level standards for analytes of interest. Elution profiles of the hydrocarbon mixtures are compared back to the initial calibration to verify the retention ranges.

11.2.4.1. A CCV for Method AK101.0 (State of Alaska) analysis includes analysis of the alkane range standard to verify retention times as well.

11.2.5. After every 20 samples (including QC and instrument blanks), or 12 hours from the time of the opening CCV, whichever is more frequent, a continuing calibration is analyzed. The continuing calibration consists of a mid-level standard differing in concentration from the opening CCV of all petroleum hydrocarbons of interest.

11.3. Low-Level Solids Analysis -- Refer to the main body of this SOP for specifics of low-level solids analysis.

11.3.1. This method may not be used for samples analyzed to support Alaska or Washington State soil investigations.

12. CALCULATIONS/DATA REDUCTION

12.1. Refer to Section 12 of the main body of this SOP for general information regarding data analysis and calculations.

12.2. Qualitative Identification

For fuels (multicomponent analytes), identification occurs when a peak pattern matching the reference standard is found within the elution window for a fuel, at a concentration above the reporting limit, or above the MDL if J flags are required. If the peaks present in the chromatogram elute within the time window of the reference fuel, but the pattern does not match the reference fuel, then the material is determined to be an “Unknown Hydrocarbon” and will be reported accordingly. See the method appendices for further information regarding fuel identification and reporting.

12.2.1. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2.2. Criteria for determining Gasoline, degraded Gasoline, and Unknown Hydrocarbons:

Presence of BTEX compounds. Native gasoline has a very high BTEX content, and degraded materials typically exhibit this as well. If the sample has been analyzed for BTEX as well as gasoline, the presence and concentration of BTEX relative to the concentration of the hydrocarbon material is a good indicator of gasoline vs. unknown hydrocarbon.

12.2.3. Reporting for ADEC Method AK101.0 (Alaska-GRO)

Peak areas between the start of n-C06 and the start of n-C10 are summed. A concentration is calculated, and the results are reported as "Gasoline Range Organics" (GRO).

12.3. Gasoline is quantitated using the area between two marker peaks as specified in Table A2. Because the methanol solvent peak appears in the chromatogram, it is not advisable to sum the entire chromatogram.

12.4. Calculating the "on-column" amount in an unknown sample using Average RRF:

Equation A6

$$C_X = \frac{A_X}{\overline{RF}}$$

Where: A_X = Area of the total ion chromatogram for the compound to be measured
C_X = Concentration of the compound being measured, ng/mL
$\overline{RF}$ = Average response factor of the compound.

12.5. Calculating the "on-column" amount in an unknown sample using a linear fit, not forced through zero. (Based on the "Target" data system).

Equation A7

$$C_X = b + \frac{A_X}{m1}$$

Where: A_X = Area of the total ion chromatogram for the compound to be measured
C_X = Concentration of the compound being measured, ng/mL
b = Constant as calculated by Target
$m1$ = First degree factor, as calculated by Target

12.6. Calculate results in samples as in Section 12 of the main portion of this SOP.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance criteria.

14. POLLUTION CONTROL

Refer to Section 14 of the main body of this SOP for information relating to pollution control.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for information relating to waste management.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035, 8000B, and 8015B.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, March 2003, Section 8000C.

16.3. Leaking Underground Fuel Tank Field Manual, State of California, October, 1989

16.4. Underground Storage Tanks Procedures Manual, Alaska Department of Environmental Conservation, December, 10, 1998; Appendix D

16.5. Alaska Department of Environmental Conservation, March 1999; Method AK101.0

16.6. Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997, Method NWTPH-Gx

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. This method is performed on a GC/MS, with surrogate values calculated using Method 8260B, and the TPH values calculated using the total area chromatogram. All of the reference methods (CA LUFT, Method 8015B, AK101, and NWTPH-Gx) are based on GC-FID as the detector and the limitations thereof.

17.3. The California LUFT Manual (October, 1989) recommended DHS Analytical Methods indicate that a 3 point calibration is sufficient. However, as many clients default to SW-846, this method defaults to the more stringent SW-846 criterion.

17.4. The California LUFT Manual (October, 1989) recommended DHS Analytical Methods require a 10%D criterion on continuing calibration standard. However, as many clients default to SW-846, this method defaults to the SW-846 criterion. It is believed that since this lab follows SW-846 criteria, and the DHS method is recommended, the 15%D criteria is sufficient to meet data quality objectives.

17.5. ADEC methodology specifies that semivolatile stock standards expire after 6 months, TestAmerica Sacramento policy and this SOP state that such standards expire after 1 year.

17.6. ADEC methodology specifies that standards and extracts are stored at –10 to –20°C. This SOP used 4 ± 2°C as the requirement. This temperature range is consistent with other SOPs at this facility.

17.7. NWPTH methodology specifies a linear regression curve fit which is evaluated through the correlation coefficient. This facility defaults to average response factor calibration, and evaluates it through the %RSD. Linear regression curve fits, while permitted in the SOP, are not commonly utilized.

17.8. ADEC methodology specifies a ≤ 25% RSD criterion for evaluating calibration curves. This SOP defaults to the more stringent SW846 criterion of ≤ 20% RSD.

17.9. ADEC methodology specifies a ≤ 25% difference criterion for the 12 hour calibration. This SOP defaults to the more stringent SW846 criterion of ≤ 15% difference.

17.10. Purge volumes greater than 5 mL (up to 25 mL) may be used as required to meet reporting limits.

17.11. The shutoff valve for the sample-measuring gas tight syringe has been omitted.

17.12. The use of a soil aliquot between 1 g and 5 g is allowed if performance criteria are met.

17.13. The method has been extended to include the use of VOA vial sampling purge and trap autosamplers.

17.14. A composite gasoline standard available from an outside vendor is recommended, rather than the gasoline composite mixtures cited in the reference methods for NWTPH-Gx.

17.15. To comply with the NWTPH-Gx (State of Washington) methodology, a correction factor must be applied to compensate for the mass of oxygenated components present in any oxygenated fuels used for the composite. Contact the vendor to obtain this value.

17.16. Method 5035 states that calibration standards for low-level solids preserved with sodium bisulfate should also have sodium bisulfate present, at approximately the same levels, in order to compensate for the effects of the preservative on analyte purgeability. At this time, TestAmerica Sacramento does not use different calibration curves for solids preserved with sodium bisulfate. All solid calibration curves omit the sodium bisulfate.

17.17. Modified purge temperature in Section 10.7.2 from 40°C to 50°C

18. ATTACHMENTS

18.1. Table A1 – Standard Analyte List

18.2. Table A2 – Volatile Petroleum Hydrocarbon Quantitation Ranges.

18.3. Table A3 – Composition and Concentration of Standard Solutions

18.4. Table A4 – LCS/MS/SD Concentrations for Aqueous Samples

18.5. Table A5 – LCS/MS/SD Concentrations for Low-Level Solid Samples

18.6. Table A6 – LCS/MS/SD Concentrations for Medium-Level Solid Samples

18.7. Table A7 – Aqueous and Medium-Level Solid Calibration Standard Concentrations (ug/L)

18.8. Table A8 – Low-Level Solid Calibration Standard Concentrations (ug/kg)

18.9. Table A9 – Calibration Standard Recipes (ICAL and CCV Standards)

18.10. Table A10 – Alkane Range Standard (5 uL spiked into 5 mL)

18.11. Table A11 – Initial Demonstration Limits

18.12. Table A12 – AK101 Recovery Requirements

Table A1				
Standard Analyte List				
Compound	CAS Number	Reporting Limit, ug/L or ug/kg		
		Aqueous	Low Soil	Medium Soil
Gasoline	39-40-0	50	500	5000
Gasoline Range Organics (GRO)	NA	50	500	5000

Table A2		
Volatile Petroleum Hydrocarbon Quantitation Ranges.		
Regulatory Method	Quantitation Range	
	Start	End
Method 8015B (GRO)	2-methylpentane	1,2,4-Trimethylbenzene
C7-C12 (CA-LUFT)	n-C07	n-C12
AK101.0[1]	n-C06	n-C10
C4-C12	n-C04	n-C12
C5-C12	n-C05	n-C12
C6-C10	n-C06	n-C10
C6-C12	n-C06	n-C12

[1] *The method specifies that the peak summing window must start at the start of the n-C06 peak and finish at the start of the n-C10 peak.*

Table A3 **Composition and Concentration of Standard Solutions**		
Components	Low-Level Working Solution Concentration, ug/mL	Medium-Level Working Solution Concentration, ug/mL
Gasoline	1000	50000

Note: "Low-Level" working solution is used for aqueous, low-level solid, and calibration standard spiking. The "Medium-Level" working solution is used for spiking medium-level samples and samples preserved with methanol.
Refer to the main body of this SOP for surrogate details.

Table A4 **LCS/MS/SD Concentrations for Aqueous Samples**			
Low-Level Solution	Working Solution ug/mL	Spike amount uL (into VOA vial)	Final concentration ug/L
Gasoline	1000	21.5	500

Refer to the main body of this SOP for surrogate details.

Table A5 **LCS/MS/SD Concentrations for Low-Level Solid Samples**			
Low-Level Solution	Working Solution ug/mL	Spike amount uL (onto 5g solid with 10 mL water)	Final concentration ug/kg
Gasoline	1000	5.0	1000

Refer to the main body of this SOP for surrogate details.

Table A6 **LCS/MS/SD Concentrations for Medium-Level Solid Samples**			
Medium-Level Solution	Working Solution ug/mL	Spike amount uL (onto 5g solid with 5 mL MeOH)	Final concentration Ug/kg
Gasoline	50,000	5.0	50,000

Refer to the main body of this SOP for surrogate details.

Table A7								
Aqueous and Medium-Level Solid Calibration Standard Concentrations (ug/L)								
Component	1	2	3	4	5	6	7	8
Gasoline (Aq)	50	100	500[1]	1,000	2,500	5,000	7,500	10,000
Gasoline (Soil)	50	100	500[1]	1,000	2,500	5,000	10,000	25,000

[1] *Designates the mid-level standard concentration used for the ICV and CCVs following the initial CCV of a sequence.*

Refer to the main body of this SOP for surrogate details.

Table A8								
Low-Level Solid Calibration Standard Concentrations (ug/kg)								
Component	1	2	3	4	5	6	7	8
Gasoline	50	100	500	1000[1]	2500	5000	7500	10000

[1] *Designates the mid-level standard concentration used for the ICV and CCVs following the initial CCV of a sequence.*

Refer to the main body of this SOP for surrogate details.

Table A9									
Calibration Standard Recipes (ICAL and CCV Standards)									
Component	Solution Conc. ug/mL	1	2	3	4	5	6	7	8
Aqueous and Medium-Level Solid Samples (uL, Spiked into 100 mL)									
Gasoline	1000	25[1]	50[1]	25[2]	50	125	250	375	--
Low-level Solid Samples (uL, Spiked into 100 mL, 10 ml is 5g equivalent)									
Gasoline	1000	2.5[1]	5.0[1]	2.5[2]	5.0	12.5	25.0	37.5	--

[1] *The Gasoline standard is a 1:10 dilution of the working 1000ug/mL standard*

[2] *This is the recipe for the CCV standard as well.*

Refer to the main body of this SOP for surrogate details.

Table A10		
Alkane Range Standard (5 uL spiked into 5 mL)		
Components	Working Solution ug/mL	Final Concentration ug/L
n-C04 (butane)	10	10
n-C05 (pentane)	10	10
n-C06 (hexane)	10	10
n-C07 (heptane)	10	10
2-Methylpentane	10	10
n-C10 (decane)	10	10
n-C12 (dodecane)	10	10
Naphthalene	10	10
1,2,4-Trimethylbenzene	10	10

Table A11		
Initial Demonstration Limits		
Analyte	Initial Demonstration Recovery Limits, %	Initial Demonstration RSD Limits, %
Gasoline	70-130	25
Additional Fuels	70-130	25

Table A12	
AK101 Recovery Requirements	
Requirement	Criteria
Surrogate recovery in samples	50-150%
Surrogate recovery in CCV, LCS/D	60-120%
LCS Recovery	60-120%
LCS/LCSD RPD	less than or equal to 20%

Title: Determination of Polycyclic Aromatic Hydrocarbons (PAH) by GC/MS-SIM Internal Standard Technique [Method 8270C]

Approvals (Signature/Date):

Victoria Nihart 5/27/14 Victoria Nihart Date Technical Manager	5/27/14 Joe Schairer Date Health & Safety Manager / Coordinator
5/28/2014 Lisa Stafford Date Interim Quality Assurance Manager	5/29/14 Robert Hrabak Date Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This method is suitable for the analysis of PAHs in solid, aqueous, and oily matrices by Method 8270C SIM analysis. Refer to Table 1 for a list of individual PAHs determined by this method.

1.2. The standard reporting limit (SRL) of this method for determining an individual compound is approximately 5ug/kg (wet weight) for soil/sediment samples, 50ng/L for groundwater samples, and 500ug/kg or higher for oily matrices samples. Refer to TALS for specific SRLs. Reporting limits will be proportionately higher for samples that require dilution.

1.3. This method is restricted to use by, or under the supervision of, analysts experienced in the use of capillary column gas chromatography/mass spectrometry. Because of the toxicity of the materials known or believed to contain PAH, certain precautions must be taken to prevent exposure to the analyst or to others.

1.4. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. The analytical method is gas chromatography combined with internal standard mass spectrometry. This entails the addition of extraction surrogates to all samples in known quantities, matrix-specific extraction of the sample with appropriate organic solvents, and analysis of the processed extract for PAHs using high-resolution capillary column gas chromatography coupled with low mass spectrometry (GC/MS) in selective ion monitoring mode (SIM). Analyte concentrations are calculated using the internal standard technique.

3. DEFINITIONS

3.1. Large Volume Injection (LVI) – An injection into a gas chromatograph that is larger than the typical 1 or 2µL injection used for hot vaporizing injectors.

3.2. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.3. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Interferences may be caused by contaminants in solvents, reagents, sorbents, glassware and other sample processing hardware that may lead to discrete artifacts and/or elevated backgrounds at the ions monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

4.2. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile gloves must be used.

5.1.2. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS**

for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. See SOP WS-OP-0001 or WS-OP-0006 for details of sample preparation equipment and supplies.

6.2. Preventive and routine maintenance is described in the "Schedule of Routine maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.3. GCMS Analytical System

6.3.1. Gas Chromatograph/Mass Spectrometer (GC/MS) – This method uses GC instrumentation manufactured by Agilent, Model 6890, or equivalent, and a Mass Spectrometer manufactured by Agilent, Model 5973 MS, or equivalent. The GC injection port must be designed for capillary columns. Splitless injection is recommended. See Table 8 for the recommended column for this analysis.

6.3.2. Mass Spectrometer - The mass spectrometer (MS) must be capable of operation in the Selective Ion Monitoring mode at a resolving power of 1 amu. Electron impact ionization must be used. The mass spectrometer must be capable of monitoring all of the ions listed in each of the four SIM descriptors (Table 9) with a total cycle time of 1 second or less.

6.3.3. GC/MS Interface

Any gas chromatograph to mass spectrometer interface may be used as long as it gives acceptable calibration response for each analyte of interest at the

desired concentration and achieves the required tuning performance criteria. To achieve maximum sensitivity, the exit end of the capillary column should be placed in the mass spectrometer ion source without being exposed to the ionizing electron beam.

6.4. Data System

A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and plot a Selected Ion Current Profile (SICP), a plot of the abundances of the selected ions versus time or scan number. Software must also be available to integrate, in any SICP, the abundance between specified time or scan-number limits.

The system utilizes ChemStation version D for data acquisition and Chrom version 2.1 for data processing.

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7. REAGENTS AND STANDARDS

7.1. See WS-OP-0001 or WS-OP-0006 for details of reagents related to sample preparation.

7.2. Stock Standard Solution

Standard solutions can be prepared from pure standard materials or purchased as certified solutions (see analyte lists in Tables 2-4). Stock standard solutions must be replaced after one year.

7.2.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampule is opened, if the standard is supplied in a sealed ampule. If a vendor-supplied date has an earlier expiration than the date from preparation, the earlier date is used.

7.2.2. Neat Standard Materials - Neat materials expire following a period of 3 years from the date of receipt, or sooner if problems such as degradation occur.

7.2.3. Working Standard Solutions - Working standards expire following a period of 1 year from the preparation date or the expiration date of the stock solution, whichever is earlier. The solution may be replaced sooner if evidence of degradation is observed.

7.3. Preparation of Stock Solutions

7.3.1. Extraction Surrogates for calibration standards - Prepare or purchase a stock solution in methylene chloride ($MeCl_2$) of the three extraction surrogates

listed in Table 2 at concentrations of 100 ug/mL.

7.3.2. Internal Standards - Prepare or purchase a stock solution in $MeCl_2$ of the five internal standards listed in Table 3 at concentrations of 2000 ug/mL.

7.3.3. Store stock standard solutions in Teflon-sealed screw-cap bottles at 0-6°C and protect from light. Stock standard solutions must be checked frequently for signs of degradation and evaporation, especially just before using them to prepare calibration standard solutions or spiking solutions.

7.3.3.1. Replace stock standard solutions every 12 months or more frequently if comparison with quality control check samples indicates a problem.

7.3.4. Calibration Standards

7.3.4.1. Prepare calibration working standard solutions by combining appropriate volumes of individual or mixed calibration standards with extraction surrogate stocks, internal standard stocks and diluting to volume with methylene chloride to obtain the solution concentrations given in Table 4. The suggested range is 50 ng/mL to 5000 ng/mL and 25 ng/mL to 1000 ng/mL for LVI . Calibration solutions must be replaced after one year from the date of preparation, or sooner if empirical data indicates the standard has undergone concentration or degradation. If one of the parent solutions has an earlier expiration date, that date must be used.

7.3.4.2. Prepare an Initial Calibration Verification (ICV) or Second Source Calibration Standard (SSCS), when available, to confirm the calibration curve. This standard will be analyzed after the initial calibration.

7.3.4.2.1. All standards must be stored at 0-6°C and must be freshly prepared if the calibration verification, internal standard, or native analyte standard indicates a problem.

7.3.5. Internal Standard Spiking Solution

Use an appropriate volume of stock solution to prepare an internal standard spiking solution in methylene chloride with the concentrations shown in Table 3. Store at 6°C or less. Spike 10 µL of the internal standard spiking solution to the final sample extract just prior to analysis to achieve a final extraction of 0.5 µg/mL.

7.3.6. Calibration Verification Standard

7.3.6.1. The calibration verification standard shall be used for column performance checks to verify peak separation, and for daily calibration checks. Solution #3 or #4 from Table 4 shall be the calibration verification standard.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Refer to SOP WS-OP-0001 for sample preservation and storage.

8.2. Extracts are stored at < -10°C until at least 30 days following invoicing.

8.3. Extracts must be analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Control Limits

The laboratory will establish historically derived recovery limits for laboratory control samples (LCS), matrix spikes (MS), and surrogates. QAPP or project specific limits may supersede, if mutually agreed upon.

9.1.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into TALS (when available) or other database so that accurate historical control charts can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.1.2. Refer to the QC Program document (WS-PQA-003) for further details of control limits.

9.2. Internal Standards

Internal standards are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard recoveries are monitored to verify that instrument performance is acceptable. Criteria for standards are delineated in Section 10. If the internal standard data indicate instrument failure, the samples may require reanalysis. If not, the impact on sample data is evaluated and the data is flagged appropriately.

9.2.1. The internal standard (IS) responses in all QC and field samples are compared to the mid-point standard of the initial calibration. Alternatively, the IS response in the samples may be compared to the daily CCV, on a per batch basis, to meet client or program requirements.

9.2.2. Any samples that do not meet the same IS criteria set forth for the CCV (Section 10.4) must be evaluated for validity. If the change in internal standard response is a matrix effect confined to an individual sample, reanalysis may not be necessary, but should be verified by the department manager or client. If the change in internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.2.3. Any IS responses outside the criteria should be noted in the case narrative.

9.3. Extraction Surrogate Spike

9.3.1. Recoveries for each of the extraction surrogates must be within the historically derived, or QAPP specific recovery limit. If the extracts are diluted > 5x the surrogates will be diluted out, and not reportable. The data flag of NC-SRD will be used to indicate this.

9.3.2. If the extraction surrogate standard recoveries are outside of the acceptable limits, the cause of the failure should be investigated. Otherwise the sample is re-extracted if sufficient sample volume is available; subject to client approval, re-extraction of a smaller sample aliquot may be appropriate to mitigate low surrogate recovery caused by matrix interferences.

9.3.3. Recommended Corrective Actions for surrogate failures in MB, LCS, or LCSD (batch QC)

9.3.3.1. Check all calculations for errors.

9.3.3.2. Verify satisfactory instrument performance.

9.3.3.3. Recalculate the data and/or reanalyze the extracts if either of the above checks reveals a problem.

9.3.3.4. If the problem persists the QC and associated samples may require re-extraction.

9.3.3.5. Review the analytical procedures with the performing laboratory personnel.

9.3.3.6. Re-extract the samples and associated QC if necessary (Example- low surrogate recovery in MB)

9.3.3.7. Document the failure in an anomaly, and any corrective action in the narrative.

9.3.4. Corrective actions for surrogate failures in field samples or MS/MSD:

9.3.4.1. Check all calculations for error.

9.3.4.2. Ensure that instrument performance is acceptable.

9.3.4.3. Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

9.3.4.4. Evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interferences demonstrated by prior analyses)

9.3.4.5. Re-extract if necessary.

9.3.4.6. Document the failure and note it on the final report.

9.4. Method Blanks

One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water, and for solid samples, the method blank is an aliquot of sodium sulfate. For air samples, the method blank may be an aliquot of XAD resin, PUF, filter, or other matrix that is representative of the sample matrix. The QC aliquots are processed in the same manner and at the same time as the associated samples. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.4.1. Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.4.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.4.5.

9.5. Laboratory Control Samples (LCS)

A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Table 5 unless otherwise specified by a client or agency.

9.5.1. If any analyte in the LCS is outside the method control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

9.5.2. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the LCS recovery is high, the samples are ND, and sample surrogate recoveries are good).

9.5.3. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.4. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples for aqueous and solid matrices, where sufficient sample volume is available. The MS/MSD is spiked with the same set of analytes as the LCS (See Table 5). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

9.6.1. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

9.6.2. If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.

9.6.3. If an MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed if required by the program or client. RPD of the LCS and LCSD are compared to the matrix spike limits.

9.6.4. The MS/MSD must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Initial Calibration

The initial calibration is required before any samples are analyzed, and then intermittently throughout sample analyses as dictated by results of the continuing calibration procedures described in this section, and after any major maintenance. The GC/MS system must be properly calibrated and the performance documented during the initial calibration.

10.2.1. GC/MS Tuning Criteria

Use a compound such as perfluorotributylamine (PFTBA) to verify that the intensity of the peaks is acceptable. If PFTBA is used, mass spectral peak profiles for m/z 69, 219, and 264 must be recorded, plotted and reported. The scan should display a minimum of ± two amu (i.e. m/z 67-71 for the m/z 69 profile) and the masses be within 0.50 amu of the target masses. The tune is verified at the beginning of every continuous sequence.

NOTE: The requirement to analyze the instrument performance check solution is optional when analysis of Polyaromatic Hydrocarbons (PAHs)/pentachlorophenol is to be performed by the Selected Ion Monitoring (SIM) technique. (USEPA CLP SOW1.2)

NOTE:The EPA CLP Functional Guidelines for semivolatile organic analysis, Section II (Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check), Item B (Objective), states "GC/MS instrument performance checks are performed to ensure adequate mass resolution, identification, and to some degree, sensitivity. These criteria are not sample-specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances. **NOTE: This requirement does not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.** "

10.2.2. GC Operating Conditions

The GC column performance must be documented during every analytical sequence. Table 8 summarizes GC operating conditions known to produce acceptable results with the column listed. The GC conditions must be established for the particular instrument by injecting aliquots of the calibration check standard. The valley height between benzo(b)fluoranthene and benzo(k)fluoranthene must be no more than ½ the height of the taller peak. It may be necessary to adjust the operating conditions slightly based on observations from analysis of these solutions. Thereafter, the calibration check standard must be analyzed daily to verify the performance of the system.

10.3. Calibration Procedure for Internal Standard Method

10.3.1. The internal standards used in this method are listed in Table 3. Use the base peak m/z as the primary m/z for quantitation of the standards, and the secondary m/z for confirmation purposes only. If interferences are noted one of the next two most intense masses may be used for quantitation.

10.3.2. Compounds are assigned to the internal standards as indicated in Table 6.

10.3.3. Using stock standards, prepare at least five calibration standard solutions, using the same solvent that is used in the final sample extract. Keep the internal standards at fixed concentrations. Table 4 lists the recommended calibration standard concentrations.

10.3.4. Calibrate the mass spectrometer response using a 2-5 µL aliquot of each calibration solution. Each solution must be analyzed once. Calculate the response factors (RFs) for each analyte:

10.3.4.1. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations 1, 2, and 3 and

verify that the criteria in Table 7 are met. **No sample analysis may be performed unless these criteria are met.**

10.3.4.2. Mean Response Factor - Use Equation 2 to calculate the mean RF for each compound (calibration standards and surrogate standards). This is the average of the five RFs calculated for each compound (one RF calculated for each calibration solution).

10.3.5. If the %RSD for any analyte exceeds 30% then a linear calibration should be tried for all analytes with %RSD > 30%. Linear or quadratic curve fits may be used. Use of 1/Concentration2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990. The "Y" intercept (listed as "b" on the ICAL summary pages) should be within ± ½(RL/ISTD). If the intercept exceeds this criterion, false positives or negatives could result. If the RL is elevated for client or project requirements, use the default RL or MDL.

10.3.6. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.3.7. The %RSD should be </=15% for DoD and AFCEE projects.

10.3.8. Initial calibration verification standard (ICV) - When available, a second source standard should be used for ICV and analyzed with the initial calibration. Each compound must be within +/- 30% of its expected value. Under the DoD QSM, the criteria are +/- 20%D for all analytes.

Corrective actions for a failed ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards
- Narrate all affected data.

10.3.9. If time remains in the 12-hour period initiated by initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.3.10. Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.4. Continuing Calibration (Calibration Check)

The calibration check standard must be analyzed at the beginning of each analysis period, or at the beginning of every 12-hour shift if the laboratory operates during consecutive 12-hour shifts.

10.4.1. Open the perfluorotributylamine (PFTBA) to verify that the intensity of the peaks is acceptable. The mass spectral peak profiles for m/z 69, 219, and 264 must be recorded, plotted and reported. The scan should display a minimum of ± two amu (i.e. m/z 67-71 for the m/z 69 profile). The analysis must produce masses within 0.50 amu of the target masses to show acceptable spectral profiles. Use the same data acquisition parameters as those used during the initial calibration. This verification is performed at the beginning of every consecutive sequence and must be performed at least once a day

10.4.2. Inject a 2-5ul aliquot of the calibration check solution into the GCMS. Use the same data acquisition parameters as those used during the initial calibration.

10.4.3. Update the retention time windows for each of the compounds based on the daily standard. The retention time for the calibration standard must not change by more than 30 seconds from the most recent calibration check if no maintenance has been performed since the last check. If the retention time shifts by more than 30 seconds and no maintenance has been performed, inspect the chromatographic system for malfunctions and make the necessary corrections. Document acceptable performance with a new initial calibration curve.

10.4.4. Calculate the response factors. Refer to Section 12 for the appropriate equations.

10.4.5. Calculate the delta RF (ΔRF) which is the Relative Percent Difference (RPD) between the daily RF and the initial calibration mean RF. The measured RFs of all analytes (native and surrogate) must be within the criteria noted in Table 7, of the mean values established during the initial calibration. If this criterion is not satisfied, assess the potential impact on the data, if any possible impact is suspected appropriate corrective action must be performed. The corrective action may include re-injection of the continuing calibration standard, instrument maintenance, or injection of a new initial calibration curve, before sample extracts can be analyzed.

10.4.6. Check the EICP areas of the internal standards for the Continuing Calibration Verification Standard (CCV) versus the mid-point standard level of the most recent initial calibration sequence. If the area changes by a factor of greater than two (-50% to +100%), the analytical system must be inspected to determine the cause of the change and corrections must be made, as appropriate. If corrections are made, the CCV must be analyzed again to verify the system is operating properly. If a system malfunction is found, the samples analyzed while the system was malfunctioning must be evaluated for potential impacts, and reanalyzed as necessary.

10.4.7. Recommended corrective actions for CCV failure:

- Re-analysis CCV.
- Instrument maintenance.
- A new initial calibration curve.

10.4.8. Once the above criteria have been met, sample analysis may begin.

11. PROCEDURE

11.1. Sample Extraction

Procedures for separatory funnel liquid/liquid extraction, sonication extraction, K-D concentration and N-Evap Concentration are described in SOP WS-OP-0001. Procedures for the extraction of air samples are described in SOP WS-OP-0006.

11.2. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.3. GC/MS Analysis

The laboratory may proceed with the analysis of QC and field samples only after demonstrating acceptable performance as specified in Section 10.

11.3.1. Calibrate the instrument as described in Section 10.

11.3.2. All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

11.3.3. Approximately 1 hour before analysis, remove the sample extracts from the freezer and the internal standard solution from the refrigerator and allow them to warm to room temperature.

11.3.4. Add 10 µL of the internal standard solution to all QC and filed samples to achieve a final concentration of 500 ng/mL. If the sample volume must be changed to achieve a desired detection limit, the internal standard solution concentration must be adjusted accordingly to achieve the target concentrations of Table 4.

11.3.5. Inject the sample extract into the instrument using the same injection technique as used for the standards.

11.3.6. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.3.7. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.3.8. Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.3.9. The presence of a given PAH is qualitatively confirmed if the criteria of Section 12.1 are satisfied. The response for any quantitation ion in the sample extract should not exceed the response of the highest concentration calibration standard. If the response exceeds the highest concentration calibration standard, the sample extract should be diluted to get the analyte response within the linear range of the calibration curve.

11.4. GC/MS Analysis - Large Volume Injection (LVI)

11.4.1. For samples being analyzed by LVI, add 5 µL of the internal standard solution to all samples and QC to achieve a final concentration of 500 ng/mL. (Final extract volume for LVI samples is 0.5 mL). If the sample volume must be changed to achieve a desired detection limit, the recovery spike solution concentration must be adjusted accordingly to achieve the target concentrations of Table 5

11.4.2. Inject a 5 µL aliquot of the sample extract on the instrument. Recommended GC/MS operating conditions are described in Table 8.

11.5. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the reporting limit and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.5.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a less dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.5.2. Reporting Dilutions

11.5.2.1. In general, the most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.5.2.2. Under DoD QSM, all valid dilutions for a sample must be reported.

11.5.3. Each time the sample extract is diluted, internal standard solution must be added to maintain a final concentration of 500 ng/mL for each internal standard component.

11.6. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at < -10°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.7. Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.7.1. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.8. Troubleshooting Guide

11.8.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the Sacrament QAM, the following daily maintenance may be performed.

- Clip column front or back as necessary.
- Replace injection port liner as necessary.
- Replace gold seal as necessary.
- Replace septum as necessary.
- Perform mass calibration as necessary

11.8.2. Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Analysis

Identification Criteria: The presence of a given PAH is qualitatively confirmed if the criteria below are satisfied.

12.1.1. The elution of the sample component must compare to within +/- 0.2 minutes of the GC retention time of the standard component in the daily calibration check.

12.1.2. The characteristic ions of the component in the sample must match standard component characteristic ions.

12.1.3. A primary and secondary ion will be monitored for all labeled and unlabeled analytes, which have a secondary ion >10% of the primary. The secondary ion will be used for qualitative identification. The presence of the secondary ion will be dependent upon the analyte concentration in the sample. The secondary ion may not be present for analytes detected at < 5x the reporting limit. Samples with concentrations of the target analyte 5-10x the reporting should have the secondary ions present at a detectable level.

12.1.4. The relative intensities of the ions should agree within +/- 30% between the standard and sample spectra.

12.1.5. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report

that identification with the appropriate footnote and proceed with quantitation.

12.1.6. If in the judgment of the analyst the separation of benzo(b)fluoranthene and benzo(k)fluoranthene is insufficient for quantitation, the pair may be reported as a total peak, based on the average response factor of the two.

12.1.7. The internal standard areas in the samples will be monitored against the mid-point initial calibration standard to evaluate possible instrument drift, but specific criteria or corrective actions would typically be defined in project specific QAPPs.

12.2. Calculations

12.2.1. Refer to policy QA-004-SAC for rules regarding significant digits in calculations. Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation.

12.2.2. Response Factors (RF) for native PAH and Surrogate Standards –Use the data obtained during initial calibration (10.2.) or continuing calibration (10.3).

Equation 1:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Area of the characteristic ion for the compound being measured

Ais = Area of the characteristic ion for the specific internal standard

Cx = Concentration of the compound being measured (µg/L)

Cis = Concentration of the specific internal standard (µg/L)

12.2.3. Mean Response Factors (RFavg) — Calculate the mean RF for each target PAH and surrogate standard, internal standard and alternate standard using Equation 2 and the RFs calculated according to Equation 1.

Equation 2:

$$\mathrm{RRF_{avg}} = \frac{1}{\mathrm{n}} \sum_{i=1}^{n} (\mathrm{RRF_i})$$

Where:

RRF = RRF calculated for calibration solution "i"

n = The number of data points derived from the calibration. The minimum requirement is a five-point calibration

12.2.4. Percent Relative Standard Deviation for Initial Calibration

Equation 3:

$$\%\mathrm{RSD} = \frac{SD}{RF_{avg}} \times 100$$

Where:

RFavg = Mean of RFs from initial Calibration for a compound.

SD = Standard deviation of RFs from initial calibration for a compound.

$$= \sqrt{\sum_{i-1}^{n} \frac{(RFi - RFavg)^2}{n = 1}}$$

RFi = RF for calibration level i

n= number of RF values used.

12.2.5. Continuing Calibration ΔRF - ΔRF is the relative percent difference (RPD) between the daily RF and the mean RF calculated during initial calibration.

Equation 4:

$$\Delta RF = \frac{RF_c - RF_{avg}}{RF_{avg}} \times 100\%$$

Where:

RFavg = Mean response factor of a given analyte

RFc = The RF of a given analyte obtained from the continuing calibration

12.2.6. Total Mass of Target PAH or Surrogate Standard in Sample – (Ms)

Equation 5:

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

C_{ex}= Concentration in extract, µg/mL

R_x = Response for analyte

R_{is}= Response for internal standard

C_{is} = Concentration of internal standard

12.2.7. Analyte Concentration in Sample, Aqueous Samples

Equation 6a:

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL)

V_o = Volume of water extracted (mL)

12.2.8. Analyte Concentration in Sample, Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis):

Equation 6b:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

Where:

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.2.9. MS/MSD percent recovery calculation.

Equation 7:

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result
S_R = Sample result
S_A = Spike added

12.2.10. Relative % Difference calculation for the MS/MSD

Equation 8:

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit (MDL)

The laboratory must generate a MDL for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each

analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

15.1. 2 milliliter autovials contaminated with methylene chloride, after extracts are analyzed. After analysis, the vials are returned to long-term storage. When released for disposal, they are moved into vial waste collection carboys. When the carboy is full, or after no more than one year, it is transferred to the waste disposal area, where the vials are run through the vial eater for disposal.

15.2. Waste methylene chloride from instrument needle cleaning process. Waste methylene chloride is collected in vials as part of the instrument operation. When full, the contents of the vial are poured into collection carboys. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment

16. REFERENCES/CROSS REFERENCES

16.1. “Handbook of Analytical Quality Control in Water and Wastewater Laboratories”, USEPA EMSL, EPA-600/4-79-019 (3/78)

16.2. CARB Method 429. Determination of Polycyclic Aromatic Hydrocarbons (PAH) Emissions from Stationary Sources. July, 1997.

16.3. Carcinogens - "Working with Carcinogens", Department of Health, Education and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug., 1977.

16.4. "OSHA Safety and Health Standards, General Industry," (29CFR1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

16.5. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

16.6. USEPA CLP SOW for Organics Analysis SOM1.2 (6/2007) Section 9.2.1

16.7. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

17. METHOD MODIFICATIONS

17.1. There are no deviations from the method.

18. ATTACHMENTS

18.1. Table 1- PAH Target Analytes and Standard Reporting Limits

18.2. Table 2- Extraction Surrogate Spiking Solution Concentration

18.3. Table 3- Internal Standard Spiking Solution Concentration

18.4. Table 4- Concentrations of the PAH's in Working GC/MS Calibration Standards

18.5. Table 4A - Concentrations of the PAH's in Working GC/MS Calibration Standards using Large Volume Analysis.

18.6. Table 5-Concentrations of Compounds in Laboratory Control Spike Samples (LCS)

18.7. Table 6- Assignment of Internal Standards for Calculating RFs and Quantiting Target PAH and Surrogate Standards

18.8. Table 7- Minimum Requirements for Response Factors for Initial and Continuing Calibrations

18.9. Table 8- Recommended GC Operating Conditions for PAH analysis

18.10. Table 9- Recommended MS Operating Conditions

19. REVISION HISTORY

19.1. WS-MS-0008/, Revision 2.5, Effective 06/06/2014

19.1.1. Inserted Section 1.2 – "The standard reporting limit (SRL) of this method for

determining an individual compound is approximately 5ug/kg (wet weight) for soil/sediment samples, 50ng/L for groundwater samples, and 500ug/kg or higher for oily matrices samples. Refer to TALS for specific SRLs. Reporting limits will be proportionately higher for samples that require dilution."

19.1.2. Inserted Section 6.2 – "Preventive and routine maintenance is described in the "Schedule of Routine maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2)."

19.1.3. Inserted Section 11.8 – "Troubleshooting Guide

19.1.4. Editorial changes.

19.2. WS-MS-0008, Revision 2.4, Effective 06/07/2013

19.2.1. Changed values in Tables 1, 3, 3A, 4, 4A, 5, 5A, 6, 7, & 8 to conform with current laboratory practices.

19.2.2. Removed Benzo(e) pyrene from analyte list in all tables.

19.2.3. Editorial changes.

19.3. WS-MS-0008, Revision 2.3, Effective 04/06/2012

19.3.1. Modifications to the SOP for adding large volume analysis procedures. Tables 3A, 4A, 5A and 8A were added for large volume analysis

19.3.2. Section 6.2.1 was modified to include instrumentation makes and models.

19.3.3. Editorial changes.

19.4. WS-MS-0008, Revision 2.2, Effective 6/25/2010

19.4.1. Editorial revisions.

19.4.2. Inserted Section 9.2

19.4.3. PAH SIM criteria changed from 25-150% to 50-200%.

19.4.4. Inserted 1000 ng/mL and 3000 ng/mL levels in Table 4.

19.5. WS-MS-0008, Revision 2.1, Effective 9/04/2009

19.5.1. Added Section 1.5, "When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated."

19.5.2. Inserted Section 10.1, “For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.”-

Table 1	
PAH Target Analytes	
Naphthalene	Pyrene
1-Methylnapthalene	Benzo(b)fluoranthene
2-Methylnapthalene	Benzo(k)fluoranthene
Acenaphthylene	Benzo(a)pyrene
Acenaphthene	Benzo(ghi)perylene
Fluorene	Indeno(123-cd)pyrene
Phenanthrene	Dibenz(ah)anthracene
Anthracene	Benzo(a)anthracene
Fluoranthene	Chrysene

Table 2	
Extraction Surrogate Spiking Solution Concentration	
Analytes	**ug/mL**
Nitrobenzene-d5	0.5
2-Fluorobiphenyl	0.5
Terphenyl-d14	0.5

Table 3	
Internal Standards Spiking Solution Concentration	
Analytes	**ug/mL**
Naphthalene-d8	50
Acenaphthene-d10	50
Phenanthrene-d10	50
Chrysene-d12	50
Perylene-d12	50

Table 4								
Concentrations of the PAH's in Working GC/MS Calibration Standaards								
Components	Solution Concentrations (ng/mL)							
	1	2	3	4	5	6	7	8
Calibration Standard								
Naphthalene	50	100	250	500	1000	2000	3000	5000
1-Methylnaphthalene	50	100	250	500	1000	2000	3000	5000
2-Methylnaphthalene	50	100	250	500	1000	2000	3000	5000
Acenaphthene	50	100	250	500	1000	2000	3000	5000
Acenanapthalene	50	100	250	500	1000	2000	3000	5000
Fluorene	50	100	250	500	1000	2000	3000	5000
Phenanthrene	50	100	250	500	1000	2000	3000	5000
Anthracene	50	100	250	500	1000	2000	3000	5000
Fluoranthene	50	100	250	500	1000	2000	3000	5000
Pyrene	50	100	250	500	1000	2000	3000	5000
Benzo(a)anthracene	50	100	250	500	1000	2000	3000	5000
Chrysene	50	100	250	500	1000	2000	3000	5000
Benzo(b)fluoranthene	50	100	250	500	1000	2000	3000	5000
Benzo(k)fluoranthene	50	100	250	500	1000	2000	3000	5000
Benzo(a)pyrene	50	100	250	500	1000	2000	3000	5000
Benzo(ghi)perylene	50	100	250	500	1000	2000	3000	5000
Indeno(123-cd)pyrene	50	100	250	500	1000	2000	3000	5000
Dibenz(ah)anthracene	50	100	250	500	1000	2000	3000	5000
Extraction Surrogate Standards								
Nitrobenzene-d5	50	100	250	500	1000	2000	3000	5000
Fluorobiphenyl	50	100	250	500	1000	2000	3000	5000
Terphenyl-d14	50	100	250	500	1000	2000	3000	5000
Internal Standard								
Naphthalene-d8	500	500	500	500	500	500	500	500
Acenaphthene-d10	500	500	500	500	500	500	500	500
Phenanthrene-d10	500	500	500	500	500	500	500	500
Chrysene-d12	500	500	500	500	500	500	500	500
Perylene-d12	500	500	500	500	500	500	500	500

Table 4A								
Large Volume Analysis (5uL) Concentrations of the PAH's in Working GC/MS Calibration Standaards								
Components	Solution Concentrations (ng/mL)							
	1	2	3	4	5	6	7	8
Calibration Standard								
Naphthalene	25	50	100	200	400	600	800	1000
1-Methylnaphthalene	25	50	100	200	400	600	800	1000
2-Methylnaphthalene	25	50	100	200	400	600	800	1000
Acenaphthene	25	50	100	200	400	600	800	1000
Acenanapthalene	25	50	100	200	400	600	800	1000
Fluorene	25	50	100	200	400	600	800	1000
Phenanthrene	25	50	100	200	400	600	800	1000
Anthracene	25	50	100	200	400	600	800	1000
Fluoranthene	25	50	100	200	400	600	800	1000
Pyrene	25	50	100	200	400	600	800	1000
Benzo(a)anthracene	25	50	100	200	400	600	800	1000
Chrysene	25	50	100	200	400	600	800	1000
Benzo(b)fluoranthene	25	50	100	200	400	600	800	1000
Benzo(k)fluoranthene	25	50	100	200	400	600	800	1000
Benzo(a)pyrene	25	50	100	200	400	600	800	1000
Benzo(ghi)perylene	25	50	100	200	400	600	800	1000
Indeno(123-cd)pyrene	25	50	100	200	400	600	800	1000
Dibenz(ah)anthracene	25	50	100	200	400	600	800	1000
Extraction Surrogate Standards								
Nitrobenzene-d5	25	50	100	200	400	600	800	1000
Fluorobiphenyl	25	50	100	200	400	600	800	1000
Terphenyl-d14	25	50	100	200	400	600	800	1000
Internal Standard								
Naphthalene-d8	500	500	500	500	500	500	500	500
Acenaphthene-d10	500	500	500	500	500	500	500	500
Phenanthrene-d10	500	500	500	500	500	500	500	500
Chrysene-d12	500	500	500	500	500	500	500	500
Perylene-d12	500	500	500	500	500	500	500	500

TABLE 5

Concentrations of Compounds in Laboratory Control Spike Samples (LCS)

Compound	Aqueous (ng/L)	Solid (ng/g)
Naphthalene	250	25
1-Methylnapthalene	250	25
2-Methylnapthalene	250	25
Acenaphthylene	250	25
Acenaphthene	250	25
Fluorene	250	25
Phenanthrene	250	25
Anthracene	250	25
Fluoranthene	250	25
Pyrene	250	25
Benzo(a)anthracene	250	25
Chrysene	250	25
Benzo(b)fluoranthene	250	25
Benzo(k)fluoranthene	250	25
Benzo(ghi)perylene	250	25
Indeno(123-cd)pyrene	250	25
Dibenz(ah)anthracene	250	25

Table 6 Assignment of Internal Standards for Calculating RFs and Quantitating Target PAHs and Surrogate Standards	
Analyte	**Internal Standards**
Compound	
Naphthalene	Naphthalene-d8
1-Methylnaphthalene	Naphthalene-d8
2-Methylnaphthalene	Naphthalene-d8
Nitrobenzene-d5 (surr)	Naphthalene-d8
Acenaphthylene	Acenaphthene-d10
Acenaphthene	Acenaphthene-d10
Fluorene	Acenaphthene-d10
2-Fluorobiphenyl (surr)	Acenaphthene-d10
Phenanthrene	Phenanthrene-d10
Anthracene	Phenanthrene-d10
Fluoranthene	Phenanthrene-d10
Pyrene	Phenanthrene-d10
Terphenyl-d14 (surr)	Chrysene-d12
Benzo(a)anthracene	Chrysene-d12
Chrysene	Chrysene-d12
Benzo(b)fluoranthene	Perylene-d12
Benzo(k)fluoranthene	Perylene-d12
Benzo(a)pyrene	Perylene-d12
Indeno(1,2,3-c,d)pyrene	Perylene-d12
Dibenz(a,h)anthracene	Perylene-d12
Benzo(ghi)perylene	Perylene-d12

Table 7			
Minimum Requirements for Response Factors for Initial and Continuing Calibrations			
Analyte	Initial Calibration +/- % RSD	Second Source Criteria ΔRF (%)	Continuing Calibration Criteria ΔRF (%)
Naphthalene	30	30	30
1-Methylnaphthalene	30	30	30
2-Methylnaphthalene	30	30	30
Acenaphthene	30	30	30
Acenaphthylene	30	30	30
Fluorene	30	30	30
Phenanthrene	30	30	30
Anthracene	30	30	30
Fluoranthene	30	30	30
Pyrene	30	30	30
Benzo(a)anthracene	30	30	30
Chrysene	30	30	30
Benzo(b)fluoranthene	30	30	30
Benzo(k)fluoranthene	30	30	30
Benzo(a)pyrene	30	30	30
Benzo(g,h,i)perylene	30	30	30
Indeno(1,2,3-c,d)pyrene	30	30	30
Dibenz(a,h)anthracene	30	30	30
Nitrobenzene-d_5	30	30	30
2-Fluorobiphenyl	30	30	30
d_{14}-Terphenyl	30	30	30

+Table 8 Recommended GC Operating Conditions for PAH analysis	
Parameter	Setting
Column	HP-5MS (or equivalent) 30m x 0.25mm id x 0.25µm film
Carrier Gas/Linear Velocity	He at 40 cm/sec
Injection Mode	Splitless
Temperature Program	60°C, hold 1.0 min, 18°C /min to 150 °C, 9°C /min. to 300°C, hold until after Benzo(g,h,i)perylene elutes (approximately 1.5 minutes)
Injector Temperature	250°C

Table 9					
Recommended MS Operating Conditions					
Parameter	Setting	Ch	Mass	Dwell (ms)	Component
Program 1					
Type	SIM	1	54	20	d_5-Nitrobenzene
High Mass	172	2	64	20	
Low Mass	54.0	3	68	20	d_8-Naphthalene
Resolution	1 amu	4	71	20	
Ionization Mode	EI+	5	82	20	Nitrobenzene-d5
Number of Channels	21	6	99	20	
Cycles/Second	1.00	7	102	20	Naphthalene
		8	112	20	
		9	127	20	Naphthalene
		10	128	20	Naphthalene Nitrobenzene-d5
		11	132	20	
		12	134	20	d_8-Naphthalene
		13	136	20	d_8-Naphthalene
		14	141	20	2-Methylnaphthalene, 1-Methylnaphthalene
		15	142	20	2-Methylnaphthalene, 1-Methylnaphthalene
		16	151	20	Acenaphthylene
		17	152	20	Acenaphthylene
		18	153	20	Acenaphthylene Acenaphthene
		19	154	20	Acenaphthene
		20	158	20	
		21	160	20	Acenaphthene-d10
		22	162	20	Acenaphthene-d10
		23	164	20	Acenaphthene-d10
		24	170	20	2-Fluorobiphenyl
		25	171	20	2-Fluorobiphenyl
		26	172	20	2-Fluorobiphenyl
Program 2					
Type	SIM	1	57	20	
High Mass	188	2	71	20	
Low Mass	80	3	80	20	d_{10}-Phenanthrene

Table 9					
Recommended MS Operating Conditions					
Parameter	Setting	Ch	Mass	Dwell (ms)	Component
Resolution	1 amu	4	85	20	
Ionization Mode	EI+	5	94	20	d_{10}-Phenanthrene
Number of Channels	10	6	141	20	
		7	165	20	Fluorene
		8	166	20	
		9	174	20	
		10	176	20	Phenanthrene, Anthracene
		11	178	20	Phenanthrene, Anthracene
		12	184	20	d_{10}-Phenanthrene
		13	188	20	d_{10}-Phenanthrene
		14	264	20	
		15	266	20	
		16	268	20	
		17	330	20	
		18	332	20	
Program 3					
Type	SIM	1	57	20	
High Mass	244	2	71	20	
Low Mass	100	3	85	20	
Resolution	1 amu	4	101	20	Fluoranthene, Pyrene
Ionization Mode	EI+	5	106	20	
Number of Channels	16	6	114	20	Benzo(a)anthracene, Chrysene
Cycles/Second		7	120	20	d_{12} –Chrysene
		8	122	20	d_{14}-Terphenyl
		9	200	20	Fluoranthene, Pyrene
		10	202	20	Fluoranthene, Pyrene
		11	208	20	
		12	212	20	d_{14}-Terphenyl
		13	226	20	Benzo(a)anthracene Chrysene
		14	228	20	Benzo(a)anthracene Chrysene
		15	236	20	d_{12} –Chrysene
		16	240	20	d_{12} –Chrysene

Table 9					
Recommended MS Operating Conditions					
Parameter	Setting	Ch	Mass	Dwell (ms)	Component
		17	244	20	d_{14} –Terphenyl
Program 4					
Type	SIM	1	57	20	
High Mass	279	2	71	20	
Low Mass	125	3	8/5	20	
Resolution	1 amu	4	126	20	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene
Ionization Mode	EI+	5	132	20	d_{12}-Perylene
Number of Channels	16	6	138	20	Indeno(1,2,3-cd)pyrene Benzo(g,h,i)perylene
Cycles/Second		7	139	20	Dibenz(a,h)anthracene
		8	146	20	
		9	252	20	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene
		10	253	20	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene
		11	260	20	d_{12}-Perylene
		12	264	20	d_{12}-Perylene
		13	276	20	Indeno(1,2,3-cd)pyrene Benzo(g,h,i)perylene
		14	277	20	Indeno(1,2,3-cd)pyrene Benzo(g,h,i)perylene
		15	278	20	Dibenz(a,h)anthracene
		16	279	20	Dibenz(a,h)anthracene
		17	284	20	
		18	288	20	
		19	292	20	

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Title: Analysis of 1,4-Dioxane by GC/MS SIM

[In-house Method]

Approvals (Signature/Date):

Victoria Nihart 4/15/14 Victoria Nihart Date Technical Manager	4/22/14 Joe Schairer Date Health & Safety Manager / Coordinator
4/15/2014 Lisa Stafford Date Interim Quality Assurance Manager	4/16/14 Robert Hrabak Date Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This method is based upon SW846 8270C, and is applicable to the determination of the concentration of 1,4-dioxane in extracts prepared from aqueous matrices.

1.2. The standard reporting limit (SRL) of this method for determining an individual compound is 1 ug/L. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.3. This method is restricted to use by, or under the supervision of, analysts experienced in the use of capillary column gas chromatography/mass spectrometry.

1.4. Refer to WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation for criteria specific for DoD projects.

2. SUMMARY OF METHOD

2.1. Aqueous samples are extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in SOP WS-OP-0001.

2.2. The analytical method is gas chromatography combined with internal standard mass spectrometry. This entails analysis of the processed extract for 1,4-dioxane using high-resolution capillary column gas chromatography coupled with low mass spectrometry (GC/MS) in selective ion monitoring mode (SIM). Analyte concentrations are calculated using the internal standard technique.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of

interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2. The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.1.2. The autovials used on the instrument contain a small amount of solvent after the analysis is completed, which can evaporate into the air through the puncture in the septa. These autovials may be collected in an open tripour or similar cup at the instrument while the analyst is removing them from the tray, but once that is complete the vials must be transferred to a waste container in a fume hood. They cannot be left out on the bench by the instrument.

5.1.3. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when working with solvent or extract containers. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.4. Exposure to chemicals must be maintained **as low as reasonably achievable**. All preparation of standards and dilutions shall be performed inside an operating fume hood. All samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Refer to SOP WS-OP-0001 for details of sample preparation equipment and supplies.

6.2. Gas Chromatograph/Mass Spectrometer System: An analytical system such as the Agilent 6890 or equivalent, complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.3. Column: 30 m x 0.25 mm I.D. 0.25-µm film thickness silicon-coated fused-silica capillary column (Agilent HP-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.4. Mass Spectrometer - The mass spectrometer (MS) use is a Agilent 5973 or equivalent, and ust be capable of operation in the Selective Ion Monitoring mode at a resolving power of 1 amu. Electron impact ionization must be used. The mass spectrometer must be capable of monitoring all of the ions listed in each of the SIM descriptors (Table 3) with a total cycle time of 1 second or less.

6.5. GC/MS Interface: HP-6890 gas chromatograph to Agilent 5973 mass spectrometer interface (or equivalent) may be used as long as it gives acceptable calibration response for each analyte of interest at the desired concentration and achieves the required tuning performance criteria. To achieve maximum sensitivity, the exit end of the capillary column should be placed in the mass spectrometer ion source without being exposed to the ionizing electron beam.

6.6. Data System: A Chemstation G17010A version 0.02.00 sp1. Windows 2000, service pack 4 (or equivalent) computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and plot a Selected Ion Current Profile (SICP, a plot of the abundances of the selected ions versus time or scan number). Software must also be available to integrate, in any SICP, the abundance between specified time or scan-number limits. The data system must provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. The laboratory utilizes Data Station/Chemstation Processing/Chrom.

6.7. Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.8. Carrier gas: Ultra high purity helium.

7. REAGENTS AND STANDARDS

7.1. See SOP WS-OP-0001 for details of reagents related to sample preparation.

7.2. Stock Standard Solution

Standard solutions can be prepared from pure standard materials or purchased as certified solutions. Stock standard solutions must be replaced after one year.

7.2.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampule is opened, if the standard is supplied in a sealed ampule. If a vendor-supplied date has an earlier expiration than the date from preparation, the earlier date is used.

7.2.2. Working Standard Solutions - Working standards expire following a period of 1 year from the preparation date or the expiration date of the stock solution, whichever is earlier. The solution may be replaced sooner if evidence of

degradation is observed.

7.3. A minimum five-point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Table 9 for typical calibration levels.

7.4. An Internal Standard solution is prepared. See Table 7 for composition of this solution.

7.4.1. Internal Standards are added to all standards and extracts to result in 10 ng injected onto the column. For example, if the volume of an extract used is 200 µL, 1 µL of a 2000 µg/mL internal standard solution would be added for a 1 µL injection.

7.5. The standards listed above should be refrigerated at ≤ 6ºC when not in use. Refrigeration at -10ºC to -20ºC may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The continuing calibration standard is stored at ≤ 6ºC.

7.6. The standards must be replaced at least once a year. Rotate them to the Hazardous Waste storage area for disposal.

7.7. Prepare an Initial Calibration Verification (ICV) or Second Source Calibration Standard (SSCS), when available, to confirm the calibration curve. This standard will be analyzed after the initial calibration.

7.7.1. All standards must be stored at 4 ±2°C and must be freshly prepared if the calibration verification, internal standard, or native analyte standard indicates a problem.

7.8. Calibration Verification Standard

The calibration verification standard shall be used for column performance checks to verify peak separation, and for daily calibration checks. Solution #3 or #4 from Table 9 shall be the calibration verification standard.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Refer to SOP WS-OP-0001 for sample bottle preservation and storage.

8.2. Extracts are stored at –10 to -20°C until at least 30 days following invoicing.

8.3. Extracts must be analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery ±3 standard deviations for surrogates, MS and LCS. Precision limits for matrix spikes/matrix spike duplicates are mean relative percent difference ± 3 standard deviations.

9.2.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is 5X or greater.

9.2.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into TALS or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3. Refer to WS-PQA-003 for further details of control limits.

9.2.4. Current control limits for surrogates, LCS, and matrix spikes may be obtained from the TALS database..

9.3. Method Blank

A method blank is prepared and analyzed with each batch of samples. The laboratory will attempt to run all samples from a preparation batch within the same sequence on the instrument. If this is not possible, samples may be run on different sequences or different instruments, if accompanied by an instrument blank as per Section 9.4. The method blank consists of reagent water for aqueous samples (Refer to SOP WS-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.
- The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low, and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.
- The DOD QSM criteria for method blanks is no analytes detected greater than ½ the reporting limit (RL). For common laboratory contaminants, it is no analytes detected greater than the RL.

9.3.1. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.2. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.3. Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4. Instrument Blank

Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5. Laboratory Control Sample (LCS)

A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS contains the analytes of interest (see Table 5), and must contain the same analytes as the matrix spike. Upon client request, additional analytes may be added to the LCS. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch. The analyst should monitor the system if non-control analytes are outside of established control limits, and perform corrective action as necessary to prevent re-occurring failures.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of

acceptable reasons for not reanalyzing might be that sample was non-detect and the LCS had high recoveries, indicating a high bias).

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.
- If a sample is re-extracted past holding time, both sets of data will be reported.

9.5.1. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same set of analytes as the LCS (See Table 5). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the matrix spike/spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed if batch precision is required by the program or client. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Surrogates

9.7.1. Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 6.

9.7.2. Corrective actions for surrogate failures in MB, LCS, or LCSD (batch QC samples)

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Reanalyze the QC sample and/or reanalyze all samples in the QC batch.

Note: Unless otherwise specified by the client, it may be possible to report qualified results if method QC surrogate recoveries are biased high and analytes were not detected in the field samples. However, all other QC requirements would have to be met and the failure would have to be noted as an NCM.

9.7.3. Corrective actions for surrogate failures in field samples or MS/MSD:

- Check calculation and instrument performance.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses);
- consult with the client in the event of low bias (as evidenced by low recoveries) in conjunction with possible matrix effect;
- re-analyze or re-prepare the affected samples;
- and document the failure and note it in the final report

Note: Some client programs require reanalysis to confirm matrix interferences. Check special project instructions for this corrective action.

9.7.4. The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to re-prepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.7.5. If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.6. If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.7. If the reanalysis does confirm the original results, the sample will be re-extracted. If the re-extraction confirms, the original analysis is reported and the data flagged as estimated due to matrix effect. If re-extraction does not confirm (all surrogates pass in the re-extraction), only the re-extraction is reported (unless the re-extraction is outside holding times, in which case both sets of results are reported.

9.8. Internal Standards

Internal standards (IS) are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard response is monitored to verify that instrument performance is acceptable. Criteria for standards are delineated in Section 10 and must be met before sample analysis can start.

Internal standards in samples are monitored for instrument drift. If the internal standard data indicate instrument failure, the samples may require reanalysis. If IS response is affected by matrix effects, the impact on sample data is evaluated and the data is flagged appropriately.

9.8.1. Any samples that do not meet the same internal standard criteria set forth for the continuing calibration as compared to the initial calibration (ICAL) must be evaluated for validity. Alternatively, the internal standards in the samples may be compared to the response of the daily CCV, on a per batch basis, to meet client or program requirements. Evaluation may include re-analysis, unless there is obvious matrix interference. If the change in internal standard response is a matrix effect confined to an individual sample (and not due to instrument drift) reanalysis may not be necessary, but should be verified by the department manager or client. Any internal standard responses outside the criteria should be noted in the case narrative. If the change in internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.9. Initial Calibration Verification Standard (ICV): When available, a second source calibration standard is analyzed with the initial calibration curve. Each compound of the ICV must be within ± 30% of its expected value. Corrective actions for failed ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate instrument conditions.
- Evaluated the initial calibration standards.

9.10. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.11. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.12. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. Summary

The instrument tune is verified using the instrument's autotune function. Then an initial calibration is performed with a minimum five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 2.

10.2. All standards and extracts are allowed to warm to room temperature before injecting.

10.3. GC/MS Tuning Verification

Using a compound such as perfluorotributylamine (PFTBA), verify that the intensity of the peaks is acceptable. If PFTBA is used, mass spectral peak profiles for m/z 69, 219, and 264 must be recorded, plotted and reported. The scan should display a minimum of ± two amu (i.e. m/z 67-71 for the m/z 69 profile). The analysis must produce masses within 0.50 amu of the target masses to show acceptable spectral profiles. The tune is verified at the beginning of every continuous sequence.

10.4. GC Operating Conditions

The GC column performance must be documented during every analytical sequence. Table 2 summarizes GC operating conditions known to produce acceptable results with the column listed. The GC conditions must be established by each analyst for the particular instrument by injecting aliquots of the calibration check standard. It may be necessary to adjust the operating conditions slightly based on observations from analysis of these solutions. Thereafter, the calibration check standard must be analyzed daily to verify the performance of the system.

10.5. Initial Calibration

The initial calibration is required before any samples are analyzed, and then intermittently throughout sample analyses as dictated by results of the continuing

calibration procedures described in this section, and after any major maintenance. The GC/MS system must be properly calibrated and the performance documented during the initial calibration.

10.5.1. Analyze each calibration standard (Table 9) and tabulate the area of the primary characteristic m/z against concentration for each compound and surrogate standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations 1, 2, and 3. The %RSD must be less than or equal to 15 % for the calibration to be acceptable. If the %RSD does not meet this criterion, evaluate the calibration using a calibration curve fit, or perform instrument maintenance and repeat the calibration, and verify that the criteria are met. **No sample analysis may be performed unless these criteria are met.**

10.5.2. Calibration curve fitting. Calibrations used to generate factors for regression equations are delineated in SOP CA-Q-S-005, Calibration Curves (general).

10.5.2.1. If the %RSD is ≤ 15% for a compound, the average relative response factor is used to quantitate that compound.

10.5.2.2. If the %RSD is > 15% for a compound, attempt a linear curve fit. Linear curve fits must have a correlation coefficient (r) of ≥ 0.995, or a coefficient of determination (r^2) of ≥ 0.990. In addition, the y intercept (listed as "b" on the Chrom/TALS ICAL summary page) must be within ±1/2(RL/ISTD). If the intercept exceeds this criterion, false positives or negatives could result. If the RL is elevated for client or project requirements, use the default RL in the calculation.

Note: The RL value must be in terms of the amount "on-column" on the instrument.

10.5.2.3. If the linear fit does not meet criteria, a quadratic curve fit may be attempted. The coefficient of determination (r^2) must be ≥ 0.990 and the y intercept (listed as "b" on the Target ICAL summary page) must be within ±1/2(RL/ISTD). If the intercept exceeds this criterion, false positives or negatives could result. In this case, use the force through zero option (per Method 8000C.) If the RL is elevated for client or project requirements, use the default RL in the calculation

Note: The RL value must be in terms of the amount "on-column" on the instrument.

10.5.3. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration2 weighting (often called 1/X2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.5.4. If time remains in the 12-hour period initiated by initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.5.5. Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.6. Initial Calibration Verification Standard (ICV): When available, a second source calibration standard is analyzed with the initial calibration curve. Each compound of the ICV must be within ± 30% of its expected value. Corrective actions for failed ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate instrument conditions.
- Evaluated the initial calibration standards.

10.7. Continuing Calibration (Calibration Check)

The calibration check standard must be analyzed at the beginning of each analysis period, or at the beginning of every 12-hour shift if the laboratory operates during consecutive 12-hour shifts.

10.7.1. Evaluate the tune as noted in section 10.3. Use the same data acquisition parameters as those used during the initial calibration. Verify at the beginning of every consecutive sequence. As this check can not be performed while the instrument is in sequence, consecutive 12 hours runs may be analyzed, providing the continuing calibration standard meets all criteria.

10.7.2. Inject a 1-2ul aliquot of the calibration check solution (a mid-level calibration standard) into the GCMS. Use the same data acquisition parameters as those used during the initial calibration.

10.7.3. The following criteria must be met for the continuing calibration to be acceptable:

10.7.3.1. The percent difference or drift from the initial calibration must be ≤ 30%.

10.7.3.2. The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.

10.7.4. Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Preparation

Samples are prepared following SOP WS-OP-0001.

11.3. Sample Analysis Procedure

11.3.1. Calibrate the instrument as described in Section 10.

11.3.2. All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

11.3.3. Add internal standard to the extract to result in 10 ng injected on column (for example, 5 µL internal standard solution in 1.0 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

11.3.4. Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.3.5. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.3.6. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.3.7. Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.4. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be targeted to be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample should be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.4.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.4.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4.3. Each time the sample extract is diluted, internal standard solution must be added to maintain a final concentration of 10 µg/mL for each internal standard component. See Table 8 for guidance as to solution volumes when diluting.

11.5. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at -10 to -20°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.6. Retention Time Criteria for Samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.6.1. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.7. Preventative Maintenance and Troubleshooting

11.7.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the TestAmericaSacramento Quality Assurance Manual (QAM), the following daily maintenance should be performed and documented.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

GC/MS Preventive Maintenance
On an As Needed Basis:
Replace septum. Clean injector port. Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Replace injection port liner when front portion of capillary column is removed. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed. Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels. Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination. Replace filaments when both filaments burn out or performance indicates need for replacement. Check mass calibration (PFTBA or FC-43). Check ion source and analyzer (clean, replace parts as needed). Check vacuum, relays, gas pressures and flows. Change oil in the mechanical rough pump. Relubricate the turbomolecular pump-bearing wick. HP 7673 Autosampler: Replace syringe.
On a Daily Basis:
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. Autosampler: fill wash bottle, dispose of waste bottle contents. Air Autosampler: Check for proper operation. Leak check system.

11.7.2. Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or

repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Analysis

Identification Criteria: The presence of a compound is qualitatively confirmed if the criteria below are satisfied.

12.1.1. The elution of the sample component must compare to within ± 0.2 minutes of the GC retention time of the standard component in the daily calibration check.

12.1.2. The characteristic ions of the component in the sample must match standard component characteristic ions.

12.1.3. A primary and secondary ion will be monitored for all labeled and unlabeled analytes, which have a secondary ion >10% of the primary. The secondary ion will be used for qualitative identification. The presence of the secondary ion will be dependent upon the analyte concentration in the sample. The secondary ion may not be present for analytes detected at < 5x the reporting limit. Samples with concentrations of the target analyte 5-10x the reporting should have the secondary ions present at a detectable level.

12.1.4. The relative intensities of the ions should agree within ± 30% between the standard and sample spectra.

12.1.5. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification with the appropriate footnote and proceed with quantitation.

12.1.6. The internal standard areas in the samples will be monitored against the daily standard to evaluate possible instrument drift, but specific criteria or corrective actions would typically be defined in project specific QAPPs.

12.2. Percent Relative Standard Deviation for Initial Calibration

EQUATION 1

$$RSD = \frac{\sqrt{\sum_{i-1}^{n} \frac{\left(RF_i - \overline{RF}\right)^2}{n-1}}}{\overline{RF}} \times 100$$

Where:

$\overline{RF}$ = Mean of RFs from initial calibration for a compound,

RFi = RF for each of the calibration levels

N = Number of RF levels

12.3. Continuing Calibration Percent Drift

EQUATION 2

$$\%\text{Drift} = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

Where:

C_{actul} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.4. Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration. Variables for the equations below include:

C_{ex} = Concentration in extract, µg/mL

R_x = Response for analyte

R_{is} = Response for internal standard

C_{is} = Concentration of internal standard

A = Intercept

B = Slope (may also be average response factor)

C = Curvature

12.4.1. Average Response Factor

If the average of all the %RSDs of the response factors in the initial calibration is < 15%, the average response factor from the initial calibration may be used for quantitation.

EQUATION 3

$$C_{ex} = \frac{R_x C_{is}}{R_{is} B}$$

12.4.2. Linear Fit

EQUATION 4

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

12.4.3. Quadratic Fit

EQUATION 5

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

12.5. The concentration in the sample is then calculated.

EQUATION 6

$$\text{Concentration, } \mu g/L = \frac{CexVt}{Vo}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.6. MS/MSD Percent Recovery Calculation.

EQUATION 7

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.7. Relative Percent Difference calculation for the MS/MSD

EQUATION 8

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8. Relative Response Factor Calculation.

EQUATION 9

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_x = Concentration of the compound being measured (µg/L)

C_{is} = Concentration of the specific internal standard (µg/L)

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for all matrices to be analyzed is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample spiked at 1–4x the MDL should be analyzed using the same procedures used to analyze samples, including sample preparation for the IDOC. The concentration of the CDOC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION PREVENTION

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner.

Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials contaminated with methylene chloride. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methylene chloride to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2. J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975).

16.3. CA-Q-S-005, Calibration Curves (general).

17. METHOD MODIFICATIONS

17.1. Modifications from Reference Method

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2. Due to retention time shifts as a result of column clipping and long-lived initial calibrations, the internal standard retention time criteria for continuing calibrations has been removed.

17.1.3. The percent drift criterion for the continuing calibration and ICV is set at less than or equal to 30%, rather than the 20% recommended by the method in the absence of CCC and SPCC compounds.

18. ATTACHMENTS

18.1. Table 1- Analytes and Standard Reporting Limits

18.2. Table 2- Suggested Instrument Conditions

18.3. Table 3- Recommended MS Operating Conditions

18.4. Table 4- Analytes in Appropriate Retention Time Order and Characteristic Ions

18.5. Table 5- LCS and MS/MSD Spiking Components and Concentrations in the Sample

18.6. Table 6- Surrogate Compounds and Spiking Levels

18.7. Table 7- Internal Standard Solution

18.8. Table 8- Dilution Guidance

18.9. Table 9- Calibration Levels

19. REVISION HISTORY

19.1. WS-MS-0011 Revision 1.1, Effective 04/18/2014

19.1.1. Updated Section 6.5 and 6.6 with current instrumentation and data acquisition equipment.

19.1.2. Replaced 'QuantIMS' with 'TALS' in Section 9.2.2.

19.1.3. Updated Section 13.3.1 – "Four aliquots of the QC check sample spiked at 1–4x the RL should be analyzed using the same procedures used to analyze samples, including sample preparation for the IDOC. The concentration of the CDOC check sample should be less than or equivalent to the LCS samples.

19.1.4. Updated Table 3 with current Instrument Operating Conditions and Table 4 with MS Operating Conditions.

19.1.5. Editorial changes.

19.2. WS-MS-0011, Revision 1, Effective 8/28//09

19.2.1. This SOP has been updated to the new TestAmerica format.

19.2.2. This SOP was created as a separate SOP for the analysis of 1,4-dioxane..

19.2.3. Changed the tuning and calibration information to reflect the move from full-scan to SIM analysis.

Table 1				
Analytes and Standard Reporting Limits				
Compound	CAS No.	Aqueous ug/L	Solid ug/kg	Air ug/sample
1,4-Dioxane	123-91-1	1.0	NA	NA

Table 2	
Suggested Instrumental Conditions	
Column	DB-5MS (or equivalent) 30m x 0.25mm id x 0.25um film
Initial Column Temperature/Hold Time	35°C for 2 minutes
Column Temperature Program	35 - 135°C at 20°C/min; 135 – 310°C at 40°C/min
Final Column Temperature/Hold Time	310°C for 4 minutes.
Injector Temperature	250°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split/splitless, in splitless mode
Sample Volume	1 µl
Carrier Gas	Helium at 40 cm/sec; Pressure = 6.77; Purge Flow = 10.0; Purge Time = 0.00 min, Flow Total = 14.2

Table 3 **Recommended MS Operating Conditions**			
Type: SIM Resolution: 1amu Ionization Mode: EI+ **Number of Channels: 11 Cycles/Second: 1.00**			
Parameter	**Mass**	**Dwell (ms)**	**Component**
WINDOW 1			
High Mass	88	50	1.4-Dioxane
Low Mass	58	50	1.4-Dioxane
WINDOW 2			
High Mass	152	50	d4-1,4 Dichlorobenzene
	150	50	d4-1,4 Dichlorobenzene
	128	50	d5-Nitrobenzene
	115	50	d4-1,4 Dichlorobenzene
	82	50	d5-Nitrobenzene
Low Mass	54	50	d5-Nitrobenzene
WINDOW 3			
High Mass	115	20	
	86	20	
Low Mass	43	20	

Table 4				
Analytes in Approximate Retention Time Order and Characteristic Ions				
	Retention Time	Primary	Secondary	Tertiary
Internal Standards				
1,4-Dichlorobenzene-d4	6.921	152	150	115
Surrogates				
Nitrobenzene-d5	8.163	82	128	54
Analytes				
1,4-Dioxane	2.273	88	58	43

Table 5			
LCS and MS/SD Spiking Components and Concentrations in the Sample			
Compound	Aqueous (µg/L)	Solid (µg/kg)	Air (µg)
1,4-Dioxane	10	NA	NA

Table 6			
Surrogate Compounds and Spiking Levels			
Compound	Aqueous ug/L	Solid ug/kg	Air ug
Nitrobenzene-d5	10	NA	NA

Table 7			
Internal Standard Solution			
Component	**Concentration in Solution µg/mL**	**Concentration in Extract µg/mL**	**ng on-column (1 µL injection)**
1,4-Dichlorobenzene-d4	2000	10	10

Table 8			
Dilution Guidance			
Dilution Factor[1]	µL of Sample Extract	µL IS Solution	µL DCM
1x (undiluted)	1000	5	NA
2x	500	2.5	500
5x	200	4	800
10x	100	4.5	900

[1] For further dilutions, make serial dilutions. In general, add 1 µL IS solution for every 200 µL DCM added to the extract. In all cases, the µL IS solution to be added assumes that the source solution (undiluted extract, previous extract dilution, etc) was already spiked with IS solution.

Table 9 Calibration Levels, Primary Standard, µg/mL							
Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Analytes							
1,4-Dioxane	1	2	5	10	20	50	100
Surrogates							
Nitrobenzene-d5	1	2	5	10	20	50	100

Title: Determination of Low-Level Volatile Organics in Ambient / Indoor Whole Air Samples Using GC/MS-Scan Mode

[Methods EPA TO-14A and EPA TO-15]

Approvals (Signature/Date):

Robert Hrabak 8/13/14 Date Technical Manager	Joe Schairer 8/13/14 Date Health & Safety Manager / Coordinator
Lisa Stafford 8/13/2014 Date Quality Assurance Manager	Robert Hrabak 8/13/14 Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the analysis of low-level volatile organic compounds (VOCs), having molecular weight in the general range of 40-200 g/mol and vapor pressure greater than 0.10 Torr at 25C and 760 mm Hg in ambient air, by gas chromatography/mass spectroscopy (GC/MS) technique. This SOP is based on the EPA TO-14A/TO-15 method specifications and is applicable to various air matrices that include ambient air and indoor air.

1.2. Target analytes and reporting limits with this SOP are listed in Attachment 1. Reporting limits will be proportionately higher for samples that require dilution.

1.3. On occasion, clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the laboratory's Quality Assurance Manual (WS-QAM) in the section that discusses Service to the Client

1.4. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. An air sample and internal standards (IS) are metered by a mass flow controller onto a cryogenically cooled trap (using either a Microscale Purge and Trap or a Cold Trap Dehydration technique described in Section 11). The trap is heated and the contents are transferred to a Tenax trap to remove water. The Tenax trap is heated and the analytes are transferred to a cryofocusing module. The cryofocuser is heated to transfer the analytes to the gas chromatographic column for separation and detection by a mass spectrometer operated in the Scan Mode.

3. DEFINITIONS

3.1. Note that "must" and "shall" in this SOP denote required activities.

3.2. Air Sample Bag: Commonly referred to as FlexFilm or Tedlar bag, in 1.0-L or 3.0-L volumes, that is constructed of proprietary material (e.g., SKC or ESS).

Note: Use of air sample bags as sample collection media constitutes a modification to the method (see Section 17.5) that is defined in the final report.

3.3. Part per billion volume to volume (ppbv or ppb v/v): Concentration expressed as part of gaseous (vapor) volume of pure target compound contained in a billion part of gaseous volume of sample.

Note: This reporting unit is NOT equivalent to the common ppb unit used in soil or water analysis.

3.4. Particulate Filter: A cylindrical stainless steel fitting containing a fritted metal disc, which is connected to the valve of a passivated canister or to the vacuum flow regulator (VFR), to prevent particulate matter from entering and damaging the passivated canister or VFR.

3.5. Passivated canister: Commonly referred to as SUMMA canister, SilcoCan, or T.O.-Can in 1.0-L, 1.8-L, 6-L, 15-L, or 33-L volumes.

3.5.1. SUMMA canister: A spherical stainless steel container, of which the interior has been specially treated by a process (SUMMA passivation), that renders all surfaces inert to VOCs.

3.5.2. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3.5.3. T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and is extensively cleaned using an ultrasonic method that ensures a high-quality, passivated surface that maintains the stability of VOCs during storage.

3.6. Standard molar volume = 24.45 L/mol at standard conditions (i.e., room temperature of 25°C and standard pressure of 1 atmosphere).

3.7. Standard pressure = 1.0 atmosphere or 14.6 pounds per square inch absolute (psia) or 0 inches of mercury or 0 pound per square inch gauge (psig), based on laboratory elevation and average barometric pressure.

Note: Full vacuum (0 psia) = -30 inches of mercury vacuum

3.8. QC section: Surrogates: Organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Although not required by the method, each client and QC sample is spiked with surrogate standards via the analytical trap. Surrogates are used to monitor method performance with each sample. Surrogates are only reported to the client by request.

3.9. Vacuum Flow Regulator: A device which, when connected to a passivated canister, regulates the flow of sample into the passivated canister so that a timed, representative sample can be obtained (also called a composite sample), as opposed to an unregulated, instantaneous sample (grab sample).

3.10. Vacuum/Pressure Gauge: Device used to measure the vacuum or pressure in a passivated canister. Units of measure range from -30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All pressure units are converted to psia (psig + 14.7 = psia).

3.11. Further definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. Gas regulators are cleaned by the manufacturer using Freon 113, a target analyte in this SOP. Before using Ultra High Purity Nitrogen (UHP N2), hydrocarbon-free air, IS mix, or target compound standard mix cylinders, each regulator should be purged with the appropriate gas.

4.2. Contamination may occur in the sampling system if passivated canisters are not properly cleaned prior to use. Passivated canisters shall not be used for the collection of samples until a batch blank analysis indicates that no target compounds are present above the RL, or a level previously agreed upon between the laboratory and the client. When more stringent canister cleaning acceptance criteria are warranted based on project-specific or regulatory requirements, and then the more stringent criteria must be used. Further information regarding the cleaning and certification of passivated canisters may be found in TestAmerica SOP WS-QA-0032. All other sampling equipment including pumps, flow controllers, and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.2.1. Passivated canisters may be batch-certified or individually-certified, depending on client request.

4.2.2. Passivated canisters will be certified-clean down to the MDL of the target analytes of interest if sample results need to be evaluated down to those limits. However, the laboratory must be provided advanced notification of the requirement.

4.2.2.1. Common laboratory contaminants like Acetone and Methylene chloride may be present above the MDL. In this instance, client approval must be received prior to sending out these passivated canisters.

4.3. Carry-over may occur when samples with high levels of contaminants are analyzed. The sample immediately following a high-level sample shall be re-analyzed if carry-over is suspected.

4.4. Air sample bags may contain low levels of target analytes.

4.5. Only compounds having both similar mass spectrum and GC retention time (RT) would be expected to interfere in the method. This situation most commonly occurs with structural isomers.

4.6. Large concentrations of water, Methane, or Carbon dioxide may limit the size of the sample aliquot that can be effectively cryo-trapped. This may elevate the RLs for samples of this type.

4.7. Matrix interferences may be caused by non-target contaminants that are present in the sample. The extent of matrix interference will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Pressurized gas equipment is used in this procedure. Be sure all valves and gauges are operating properly and that no equipment is over-pressurized. After changing cylinders, check all gas line connectors for leaks, with soapy water. Release of high pressure gas can cause rapid suffocation.

5.1.2. This analysis may require transfer of the sample from an air sample bag to a canister (Section 11.2). Because of the flexible nature of the air sample bag, insertion of the syringe needle into the bag may cause the septa to flex or the shaft of the syringe needle may flex within the septa. This may allow sample to escape along the shaft of the needle. All samples being manually removed from an air sample bag and transferred into a canister must be handled inside a fume hood with chemical protective gloves, lab coat, and safety glasses.

5.1.3. When pressurizing canisters or changing cylinders, face shield must be worn over safety glasses.

5.1.3.1. Passivated canisters should never be pressurized over 40 psig.

5.1.4. Pressurized gas cylinders must be securely retained. The use of a face shield is required when changing regulators.

5.1.5. Air sample bags should not be pressurized, as seam splitting will result.

5.1.6. The preparation of standards and reagents will be performed in a fume hood with the sash set at the level indicated on the side of the hood.

5.1.7. Both the GC and the MS contain elevated temperature zones. These zones must be cooled prior to an analyst or technician working on the instrument.

5.1.7.1. Temperature-appropriate gloves must be worn when working with hot or cold items.

5.1.8. Latex and vinyl gloves provide no protection against organic solvents. Nitrile or similar gloves must be used.

5.1.9. The effluents from the sample splitters for the GC and the roughing pumps for the MS must be vented to a fume hood or at a minimum, must pass through a charcoal filter.

5.1.10. The MS is under deep vacuum and must be brought to atmospheric pressure before working on the source.

5.1.11. Due to high voltage risk, power to the GC and/or MS must be turned off or disconnected before work can be done on the instrument.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Helium	Simple Asphyxiant	NA – Keep oxygen levels at 19.5%	Oxygen Deficient atmosphere may cause headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting and depression of all the senses.
Liquid Nitrogen	Simple Asphyxiant Cryogenic liquid	NA – Keep oxygen levels at 19.5%	Oxygen Deficient atmosphere may cause headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting and depression of all the senses. Contact with skin may cause frostbite-changes in skin color to white or grayish-yellow.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Instrumentation

6.1.1. Gas chromatograph – capable of sub-ambient temperature programming and electronic pressure control (Hewlett Packard 6890 or equivalent).

6.1.2. Mass-selective detector – equipped with computer and appropriate software (Hewlett Packard 5973/5975 or equivalent with Chemstation data acquisition software).

6.1.3. Sample concentrator – equipped with a cryogenic trap and appropriate systems for the control of moisture (Entech 7100 or equivalent).

6.1.4. Chrom version 2.1 data processing software.

6.2. Supplies

6.2.1. Chromatographic grade stainless steel or nickel tubing and stainless steel plumbing fittings.

6.2.2. Chromatographic column – Rtx-Volatiles, 0.32 mm ID, 1.5 µm df, 60 m length, methyl polysilicate liquid phase (Restek Corporation or equivalent).

6.2.3. Transducer and process meter capable of measuring 0 psia to 50 psia, for preparing standards (Ashcroft Digital Vacuum/Pressure Gauge or equivalent).

6.2.3.1. The process meter must be calibrated quarterly, at a minimum, against the master gauge.

6.2.4. Pressure regulators for carrier gas and standards – 2-stage, stainless steel diaphragm (single stage acceptable for standards).

6.2.5. Stainless steel vacuum/pressure gauge capable of measuring from -30 inches of mercury to 40 psig (Span Instruments or equivalent).

6.2.6. Air sample bags and passivated canisters used for the preparation of standards and the dilution of samples.

6.2.7. Screen can for preparation of method blanks. This is a cleaned canister certified to be free of analytes at levels greater than or equal to the MDL or levels specified by client or program requirements, whichever is appropriate to the samples being analyzed.

6.2.8. Gas-tight syringes of various sizes (Hamilton or equivalent).

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP N_2 – used for Method Blanks and preparing dilutions of samples and standards

7.1.2. UHP Helium – used as the gas chromatograph carrier gas

7.1.3. Pressurized air source for Entech 7100 heater gas

7.1.4. Liquid N_2

7.1.5. Distilled or NanoPure water

7.2. Standards

7.2.1. Gas calibration stock standards containing the target compounds, at a nominal concentration of 1 part per million volume/volume (ppmv or ppm v/v), are purchased from NIST-approved vendors or prepared from neat in passivated canisters. Suppliers are required to provide certification of the analyte concentrations.

7.2.2. IS and surrogate stock standard mix (see Attachments 6 and 7, respectively), at a concentration of 300 ppbv, are purchased from NIST-approved vendors. Suppliers are required to provide certification of the analyte concentrations.

7.2.2.1. The surrogate mix is also used to tune the mass spectrometer.

7.3. Standard Preparation

Static dilutions and other standard preparation activities are performed in accordance with TestAmerica SOP WS-QA-0017.

7.3.1. Static dilutions of the stock standard gas mixtures are made in 6- or 15-L passivated canisters to create working standards. A high precision vacuum gauge is flushed with UHP N2 and attached to the top valve of a clean, evacuated passivated canister, and the absolute pressure is recorded.

7.3.1.1. Distilled or NanoPure water (50 µL) is added to calibration standards prior to mixing.

7.3.1.2. The IS mix does not contain water.

7.3.2. Depending on the concentration of each stock standard gas mixture, a particular pressure of each is added to the passivated canister to achieve the

desired concentration in the working standard.

7.3.2.1. As an example, the daily working standard, at a nominal concentration of 10 ppbv, is created by adding 5.0 psia of the 100 ppbv standard mix and adding UHP N_2 to the passivated canister to achieve a final pressure of 50 psia.

7.3.2.2. Care should be taken to flush each regulator and transfer line with standard prior to transfer to the passivated canister.

7.3.3. Detailed preparation of each standard is recorded in the Laboratory Information Management System (LIMS) reagent module.

7.3.4. Other preparation techniques may be used to obtain the desired standard concentration, provided these techniques do not compromise the integrity of the standards used, and that the details of the preparation are properly documented in the LIMS.

7.4. Expiration dates for standards and reagents are based on vendor specification. If no vendor expiration date is assigned, the laboratory assigns an expiration date of two years from the date of receipt. Refer to TestAmerica SOP WS-QA-0017 for further information on standards and expiration dates. Expiration dates must be documented on the gas cylinders.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample container, preservation techniques, and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Passivated Canister	2000 mL	None	30 days	EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air
Passivated Canister	2000 mL	None	30 days	Advisory – Active Soil Gas Investigations, April, 2012 (DTSC, LARWQCB, and SFRWQCB)
Air Sample Bag	500 mL	None	72 hours	N/A

8.2. Passivated canisters used for sample collection must be certified clean (< RL or program specific limit). Canisters are cleaned in accordance with TestAmerica SOP WS-QA-0032. Filters (e.g., 7-micron or 2-micron) should be placed on the inlet of the canister to protect the valve from particulates.

8.3. Samples should be shipped at room temperature, in packaging suitable to prevent puncture and exposure to light.

8.4. If air sample bags are to be shipped by aircraft, they should be filled about 75% full to allow for expansion during shipment.

8.5. The pressure of a passivated canister should be recorded before and after sample collection in the field to help detect canister leakage and document proper sampling.

8.6. Samples are stored at room temperature.

8.7. Samples should be protected from extreme temperatures.

9. QUALITY CONTROL

9.1. Batch

A batch is defined as a set of up to 20 client samples of the same matrix processed using the same procedures and the same lot(s) of reagents within the same time period. A batch must contain a Laboratory Control Sample (LCS) and a Method Blank, but they do not count towards the maximum 20 samples in a batch.

9.1.1. In some cases, an LCS Duplicate may be required by a client or program to provide batch precision. In that instance, the acceptance criteria and corrective actions appropriate for the LCS are applied.

9.1.2. Rerun of the same client sample is counted as part of the 20 in a batch (i.e., a client sample analyzed twice in the same batch must be counted as two client samples).

9.1.3. Field quality control (QC) samples (e.g., trip blanks, equipment blanks, and field duplicates) count as client samples; therefore, they add to the batch count.

9.1.4. Laboratory QC samples, including duplicates and clean canister blanks (screen cans), do not add to the batch count.

9.1.5. The batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

9.1.6. Refer to the laboratory's QC Program document (WS-PQA-003) for further details of the batch definition.

9.2. Laboratory Control Sample– For each batch, an LCS must be analyzed. The LCS is analyzed after the calibration standards and before the Method Blank and client samples. The LCS is spiked with the target analytes in Attachment 1, from which a sub-set may be reported..

9.2.1. Refer to the QC Program document (WS-PQA-003) for details on the requirements for LCS composition.

9.2.2. If any analyte is outside established recovery and precision control limits, or any surrogate is outside the established recovery control limits, the system is out of control and corrective action must occur. Corrective action typically includes reanalysis of the batch.

9.2.3. If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable reasons for not reanalyzing include evaluation of sporadic marginal exceedances (ME), or an elevated recovery (indicating a high bias) with samples non-detect for the failing analyte. Refer to the QC program document (WS-PQA-003) for more details regarding evaluation and acceptance of out of control LCS data.

9.2.4. Exceedance outside the ME limits require corrective action, regardless of whether the associated result is positive or ND. See Section 9.2.6.

9.2.4.1. For failures that exceeded the ME at the high end, the ND analyte may be flagged and reported only if the program or project-specific requirements allow.

9.2.4.2. For all other ND results that failed the ME requirements, the client must approve to flag and report the data since the nonconformance does not meet the NELAC or TNI Standard.

9.2.5. All data reported with out of control LCS values will be flagged by the LIMS. Analysts shall also file a nonconformance memo (NCM) within the LIMS detailing why the data is reported, and any corrective actions performed.

9.2.6. Corrective actions to occur before batch reanalysis:

9.2.6.1. Evaluate the analytical run for errors and anomalies. Re-analyze the LCS.

9.2.6.2. Consult the troubleshooting guidelines in Section 11.8. Evaluate the instrument status and perform maintenance.

9.2.6.3. Re-analyze the continuing calibration verification (CCV) standard and LCS, or recalibrate.

9.2.7. Current LCS control limits are stored in the LIMS. Control and ME limits are subject to change based on periodic evaluation of LCS control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.3. Method Blank

9.3.1. For each batch, an acceptable Method Blank must be analyzed. The Method Blank is analyzed after the calibration standards and LCS and prior to client samples. The Method Blank is a 6-L screen can (Section 6.2.7) humidified with 50 µL of Distilled or NanoPure water and then pressurized to 40 psia with UHP N2.

9.3.2. If a method blank is requested to be analyzed using an air sample bag similar to which samples are collected the client is required to submit a bag for that purpose. The laboratory does not maintain an inventory of air sample bags, therefore, Method blanks in bags is by client request only.

9.3.3. The Method Blank must <u>not</u> contain any analyte of interest ≥ RL (or ≥1/2 RL, as dictated by the QSM or project-specific requirements), except common laboratory contaminants, (Section 9.3.2). Otherwise, the Method Blank is further evaluated and corrective actions must be performed, as stated below. See troubleshooting guidelines in Section 11.8.

9.3.3.1. Re-analyze the Method Blank once to determine if an error or an anomaly occurred during sample analysis. If the re-analysis is acceptable, then the Method Blank can be considered in control.

9.3.3.2. If there are no results greater than the RL in the samples or if the results in the samples are greater than 10X the Method Blank level, the data may be reported with qualifiers. In this case, the elevated Method Blank result is not believed to impact data quality. The anomaly must be reported in an NCM.

9.3.3.3. If there are results greater than the RL in the samples and if these results are less than 10X the Method Blank level, the samples must be re-analyzed.

9.3.3.3.1. If re-analysis is not possible due to limited sample volume or other constraints, the Method Blank is reported and all associated samples are flagged. The client must be consulted. The anomaly must be reported in an NCM. The laboratory Project Manager

(PM) must record the client's decision in the NCM.

9.3.4. If the analyte detected in the Method Blank is a common laboratory contaminant (Methylene chloride, Acetone, and 2-Butanone), the data may be reported with qualifiers if the concentration of the analyte is less than 5X times the RL. Otherwise, corrective actions, as stated in Section 9.3.1, must be performed. The anomaly must be reported in an NCM.

9.3.5. If surrogates are a project-specific requirement, then the Method Blank must have acceptable surrogate recoveries. If surrogate recoveries are unacceptable, the data must be evaluated to determine if the Method Blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-analysis of the Method Blank and affected samples must be performed.

9.4. Surrogate Standards.

Surrogates are not a method requirement. The laboratory routinely adds surrogates to all QC and client samples via the analytical trap and will report these results only if defined in a project/contract or at client's request. The surrogate compounds used in this SOP are listed in Attachment 7.

Surrogate recoveries in QC and client samples may be assessed to ensure that recoveries are within laboratory control limits. If any surrogate is outside these limits and if surrogates are a project-specific requirement, the following corrective actions must be performed:

- Check all calculations for error.
- Ensure that instrument performance is acceptable. See troubleshooting guidelines in Section 11.8.
- Re-analyze the QC/client sample.

9.4.1. It is only necessary to re-analyze a client sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst has reason to believe that the repeated out of control results are due to problems other than matrix effect.

9.4.2. If re-analysis is not possible due to limited sample volume or other constraints, the surrogates are reported with a flag. The client must be consulted. The anomaly must be reported in an NCM. The nature of the matrix interference must be noted in the NCM. The PM must record the client's decision in the NCM.

9.4.3. Current surrogate control limits are stored in the LIMS and are subject to change based on periodic evaluation of surrogate control charts by Quality

Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.5. Internal standard (IS)

IS compounds are added to each calibration standard, LCS, Method Blank, and client sample via the analytical trap. IS compounds are monitored for each shift by comparing the IS areas and retention times in each client and QC sample against those of the associated CCV standard. The IS compounds used in this SOP are listed in Attachment 6.

9.5.1. IS evaluation criteria for the initial calibration (ICAL) may be found in Section 10.3.8.

9.5.2. For all other QC and client samples, IS areas are considered acceptable if they fall between 60% and 140% (for TO-15) or –50% and 200% (for TO-14A Low-level) of the CCV IS areas. The RTs are considered acceptable if they fall within ±20 seconds (±0.33 minutes) of the IS RT of the associated CCV.

9.5.3. Any QC or client sample exceeding the acceptance criteria above must be re-analyzed. If the IS fails upon re-analysis, the failure must be documented in an NCM. All corrective actions performed must also be documented in the NCM.

9.6. Sample Duplicate Analysis

9.6.1. A client sample duplicate is analyzed and reported with the batch when requested by the client.

9.6.2. The acceptance criteria for the duplicate analysis is an RPD ≤ 25 for target analytes detected >5X the RL. No criteria are established for duplicate results <5X the RL.

9.7. Calibration standards and other QC samples (e.g., BFB, LCS, Method Blank, etc.) may not be analyzed more than twice without documented corrective action. If the initial run fails acceptance criteria, re-inject the calibration standard or QC sample. If second run passes, analysis may proceed. Otherwise, conduct instrument maintenance or perform corrective action. Completely document failure, corrective action performed, and return to control in the Instrument Maintenance Logbook. Section 11.8 lists troubleshooting guidelines.

9.7.1. Refer to WS-PQA-021 for corrective actions specific to DoD programs.

10. CALIBRATION

For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.1. Initial/Daily Tuning of the Instrument

10.1.1. After a successful autotune per manufacturer's recommendations, each instrument is manually tuned using perfluorotributylamine (PFTBA) so that mass-to-charge ratio (m/z) 69 is 100%, m/z 131 is approximately 34%, and m/z 219 is approximately 36%. The width and axis parameters are set using the routines in the software. This initial tune should remain stable for extended periods of time, and retuning with PFTBA should not be necessary every day.

10.1.2. At the beginning of each 24-hour shift, prior to any analytical runs, it must be verified that the GC/MS system meets acceptable tune performance criteria. This is done through the analysis of 50 ng of 4-bromofluorobenzene (BFB); the acceptance criteria are listed in Attachment 4 (for TO-14A) or in Attachment 5 (for TO-15).

10.1.2.1. Using the BFB method in the cryotrap software, the IS mixture volume required is 50 ng of BFB on column (currently 28 mL of 250 ppbv standard).

10.1.2.2. An alternate way to load 50ng BFB is to use 100mL of a 10ppbv IS/SURR mix and 24mL of a 250ppbv IS/SURR mix.

10.1.2.3. The mass spectrum of BFB must be acquired using the peak apex and the scans immediately before and after the apex and averaged. A background subtraction is applied using a scan prior to the elution of BFB.

10.1.3. If any of the key ions fail the ion abundance criteria listed in the attachments, the system is considered out of tune and any subsequent sample/standard analysis shall be considered unacceptable. The BFB must be re-analyzed and re-evaluated. If the BFB continues to fail, the GC/MS system must be evaluated. See troubleshooting guidelines in Section 11.8.

10.1.3.1. Adjustments to the mass axis calibration, the electron multiplier voltage, or other tune parameters may be required. All parameter changes must be recorded in the Instrument Maintenance Logbook.

10.1.4. BFB tunes may be analyzed more frequently depending on documented client requirements.

10.2. Initial Calibration

10.2.1. Instruments are calibrated at initial setup and as needed thereafter, and at least annually.

10.2.2. An ICAL curve consisting of a minimum of five points is analyzed to determine the linear working range of the analytical system for each compound. An average response factor (RF), or sometimes called the relative response factor (RRF), and the percent relative standard deviation (%RSD) are calculated for each target analyte using the equations in Section 12.5.

10.2.3. The ICAL is considered acceptable if the calculated %RSD for the RF (or RRF) for each analyte not listed in Section 10.3.4 is <30, with at most two exceptions up to a limit of 40%.

10.2.4. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %RSD limit up to 55:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.2.5. Linear calibration using least squares regression may be used with the appropriate number of calibration points. Details regarding its use and the calculations involved may be found in TestAmerica Corporate QA SOP CA-Q-S-005. This Corporate SOP must be consulted prior to using this curve. The analyst must read and understand the topics regarding Forcing Through Zero and Curve Weighting.

10.2.6. If the ICAL acceptance criteria are not met, corrective action (documented in the Instrument Maintenance Logbook) must be performed and a new ICAL generated. See troubleshooting guidelines in Section 11.8.

10.2.7. The nominal concentrations of the ICAL standards are typically 0.20, 0.30, 0.40, 0.80, 2.0, 4.0, 8.0, 20, and 40 ppbv, but these may vary depending on the certified mix used to prepare the standards or the volume trapped. The low standard must be at or below the RL. The standards are analyzed by preparing stock standards at the required concentration or by varying the trapped volume of the working standards from the default volume of 250 mL. For example, the 0.20, 0.40, and 0.80 ppbv standards are analyzed by trapping 62.5, 125, and 250 mL, respectively, of a 0.80 ppbv working standard.

10.2.7.1. At times, the default volume may be changed, depending on required instrument sensitivity for a project.

10.2.8. Internal Standards in the ICAL

10.2.8.1. The IS response at each calibration level must fall between 60% and 140% (for TO-15) or –50% and 200% (for TO-14A) of the IS response in the mid-point calibration standard. The mid-point standard is normally 8ppbv.

10.2.8.2. The RT shift for each of the IS at each calibration level must be within ±20 seconds (0.33 minutes) of the RT of the IS in the mid-point calibration standard.

10.2.8.3. Any calibration level exceeding the above acceptance criteria must be re-analyzed.

10.2.9. The analyst may elect to drop points from the calibration curve to improve subsequent quantitation, in accordance with Policy CA-T-P-002, Selection of Calibration Points.

10.3. Essential components for ICAL evaluation

10.3.1. The signal-to-noise (S/N) in the low point of the ICAL must be ≥ 2.5:1 for the compound to be considered valid at that level. Evaluate the EICP for the S/N determination.

10.3.2. Qualitative compound identification criteria must be met for all calibration levels; (primary and secondary ions >10% must be present in each standard level. Consult the technical director or QA for clarification.

10.3.3. Check retention times for isomers to ensure correct peak assignment.

10.3.4. Check co-eluting peaks for the proper reference spectrum.

10.3.5. The ‘re-fit’ or ‘read-back’ for each point of the calibration curve is evaluated for the % error. The guideline is ≤ 20% for each level of the curve. This can be observed in the calibration summary in Chrom for each compound.

10.4. Initial Calibration Verification (ICV)

10.4.1. Each new ICAL must be verified using a second-source standard.

10.4.2. Since the regulatory agencies have not provided guidance on second-source verification, the ICV is considered acceptable if the %Recovery for each analyte not listed in Section 10.4.3 is 70–130%, with at most <u>two</u> exceptions

up to a limit of 60–140%.

10.4.3. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %Recovery of 45–155:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.4.4. For samples analyzed in the same batch where the ICAL and ICV were analyzed, every time an allowed exception to the 70–130% Recovery ICV criteria is used, the sample results (whether J-value, ND, or positive) for the affected analyte must be flagged and explained in an NCM.

10.4.5. If the ICV acceptance criteria are not met, the following corrective actions must be performed. See troubleshooting guidelines in Section 11.8.

10.4.5.1. Rerun the second-source check standard.

10.4.5.2. Re-prepare or acquire a new standard.

10.4.5.3. Evaluate instrument conditions.

10.4.5.4. Regenerate a new ICAL.

10.4.6. Due to the limited availability of second-source manufacturers for the air standard mixes and some neat compounds, the following options may be considered as second-source:

10.4.6.1. Different certified lot from the same manufacturer.

10.4.6.2. Same certified lot from the same manufacturer but the stock standard used for the second-source is prepared by an analyst other than the one who prepared the stock standard used for the ICAL. This option is only allowed if the program or project-specific requirements allow.

10.5. Continuing Calibration Verification

10.5.1. Unless the QC batch follows a new ICAL and an ICV, for every 24 hours of operation, a CCV standard is analyzed to verify the ICAL average RF. The %D of the CCV RF from the ICAL average RF is calculated for each target analyte using the equation in Section 12.5.4.

10.5.2. The CCV is considered acceptable if the %D for each analyte not listed in

Section 10.5.3 is ±30.

10.5.3. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %D ±55:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.5.4. Any time an allowed exception to the ±30%D CCV criteria is used, the sample results (whether J-value, ND, or positive) for the affected analyte must be flagged and explained in an NCM.

10.5.5. The following NELAC requirements (NELAC Quality Systems, June 5, 2003, 5.5.5.10e, page 217 of 324) and TNI Standard EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2e, page 92) apply when the CCV acceptance criteria are not met.

10.5.5.1. If routine corrective action procedures fail to produce a second consecutive (immediate) CCV within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive CCVs, or a new ICAL must be generated.

10.5.5.2. When the acceptance criteria for an analyte in the CCV are exceeded high (i.e., high bias), and the analyte was ND in the associated samples, the ND analyte may be reported with a flag. An NCM must be generated and the high bias discussed in the case narrative of the final report.

10.5.5.3. When the acceptance criteria for an analyte in the CCV are exceeded high (i.e., high bias), and the analyte was detected at a positive hit in the sample, the sample must be re-analyzed after a passing CCV or after a new ICAL has been established, evaluated, and accepted.

10.5.5.4. When the acceptance criteria for an analyte in the CCV are exceeded low (i.e., low bias), sample results may be reported if they exceed a maximum regulatory limit/decision level (if known). Otherwise, the affected samples must be re-analyzed after a passing CCV or after a new ICAL has been established, evaluated, and accepted. An NCM must be generated and the low bias discussed in the case narrative of the final report.

10.5.6. CCVs may be analyzed more frequently depending on documented client requirements.

10.6. Calibration standards and other QC samples (e.g., BFB, LCS, Method Blank, etc.) may not be analyzed more than twice without documented corrective action. If the initial run fails acceptance criteria, re-inject the calibration standard or QC sample. If second run passes, analysis may proceed. Otherwise, conduct instrument maintenance or perform corrective action. Completely document failure, corrective action performed, and return to control in the Instrument Maintenance Logbook. Section 11.9 lists troubleshooting guidelines.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Preparation – Air Bag Samples

11.2.1. For air sample bag samples, the air sample bag is checked for damage and is analyzed as received.

11.2.2. Air sample bags are analyzed directly from the bag or transferred to an evacuated can within 72 hours of sampling.

11.2.2.1. If the entire bag is transferred to a can, the bag needle valve septum is pierced with a needle attached to a 1-L or a 6-L evacuated can, and the entire contents transferred.

11.2.2.2. If only a portion of the bag is to be transferred, a measured aliquot of the bag is transferred via a clean syringe through a septum attached to the top of a 1-L or a 6-L humidified can.

11.2.3. After transfer, the can is then pressurized to a positive pressure and the pressure is recorded.

11.3. Sample Preparation – Canister Samples

11.3.1. For passivated canister samples, the initial pressure is checked by attaching the process meter line connector to the passivated canister. The process meter line connector must be rinsed before use, with the pressurization gas (UHP N2 or UHP He, if requested) by physically holding it against the gas outlet and flushing for 10 seconds, as this avoids possible carry-over concerns from high concentration samples. With the process meter line connector attached, the passivated canister valve is opened briefly and the pressure is recorded. If the pressure is less than 6 psig, the passivated canister is pressurized to 10 psig with the pressurization gas. The initial and final pressure must be recorded in the Canister Pressurization Logbook (see Attachment 11) and in the individual Canister Field Data Record.

11.3.1.1. Samples received above ambient pressure (14.6 psia) do not require pressurization unless additional volume is needed to perform multiple analyses. If samples are received below ambient pressure, UHP N_2 should be added. The default final pressure is 24 to 26 psia, however, the final pressure should be above ambient but not more than three times the initial pressure.

11.3.2. When the passivated canister vacuum/pressure is increased, a dilution factor (DF) is calculated and is applied to results. The calculation is provided in Section 12.5.5.

11.3.3. Passivated canisters received as trip blanks (without sample collected) are pressurized to 24-26 psia. These samples are considered to have a DF =1.0.

11.4. Sample Screening

Samples are screened to check for contamination before analysis or if suspected to contain significant contamination, using a GC/MS. Screening is performed to determine a proper dilution or the optimum volume of sample for the calibrated range, and to prevent overloading the analytical instrument. The screening instrument is generally calibrated at a single-point for common analytes of interest. The sample screen data are stored in TALS within the Chrom module.

11.5. Tuning

Refer to section 10.2 for details regarding instrument tuning.

11.6. Calibration

11.6.1. Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

11.6.2. Refer to Sections 10.3 through 10.5 for details regarding instrument calibration.

11.7. Sample Analysis

11.7.1. The calibration standards and the sample QC are analyzed in the same manner as client samples. After the calibration standards are analyzed and evaluated (Sections 10.3 through 10.5), the LCS is analyzed and evaluated (Section 9.2), and then the Method Blank is analyzed and evaluated (see Section 9.3), all prior to client sample analysis.

11.7.2. Each passivated canister is attached to the autosampler and recorded in the instrument sequence. A sequence is created in the GC/MS software to prepare the instrument for data acquisition. The sequence information controls the GC/MS method, data file creation, sample parameters, and report output. A second sequence must be created in the autosampler control software to control the sampling process such as line position, sample volume, trap temperatures, flow rates, and times. The sequence is verified by another analyst. This analyst verifies the autosampler sequence, port position, chem. station sequence, and Chrom worklist. The analyst annotates in the instrument maintenance log if the sequence has been verified.

11.7.3. The valves are opened on all passivated canisters and the autosampler and GC/MS sequences are started.

11.7.4. The pressure DF must be compensated for by trapping more than the default volume. For example, a sample received at 12.0 psia and pressurized to 24.6 psia has a pressure DF of 2.05. If the default volume is 250 mL, then 510 mL should be trapped. The recorded volume is rounded to three significant figures.

11.7.5. A sample that requires only a small dilution can be analyzed by trapping a volume less than the standard volume. The minimum volumes that can be trapped are 10 mL for an Entech 7200 and 20 mL for an Entech 7100. The maximum volume that can be trapped is three times the default volume. Larger dilutions may require analysis using different methodology.

11.7.6. Sample dilutions may also be performed by transferring an aliquot of the sample (originally from either an air sample bag or a passivated canister) into an air sample bag and filling it up to volume or by removing an appropriate amount of the original pressurized sample from a passivated canister and then re-pressurizing to approximately 24 to 26 psia or no more than 3x the initial pressure with UHP N_2. Serial dilutions can be performed, as necessary.

11.7.6.1. Air sample bag dilutions can be used for reporting analytes that exceeded calibration range in the original analysis, but not as a reportable analysis for all other analytes due to the low-level contamination inherent in the air sample bag.

11.7.6.2. When air sample bag dilutions are performed, the syringe used for transferring sample must be fitted with a valve (e.g., Luer-Lok) that allows the syringe contents to be isolated from the room air during transfer between containers.

11.7.7. For routine analysis, either the Microscale Purge and Trap or the Cold Trap Dehydration technique is used.

11.7.7.1. For Microscale Purge and Trap technique, the autosampler will follow the sequence of events below (parameters may be modified based on instrument performance):

11.7.7.1.1. Glass Bead trap (Module 1) is cooled to -150C

11.7.7.1.2. Internal standard is trapped

11.7.7.1.3. Sample is trapped

11.7.7.1.4. Tenax trap (Module 2) is cooled to -15C and the Glass Bead trap is heated to 10C. Any remaining sample is transferred to Module 2 by passing UHP Helium through Module 1. Conditions may vary based on instrument performance. This step is designed to remove water from the sample.

11.7.7.1.5. When GC is ready, the cryofocuser (Module 3) is cooled to –170C. Module 2 is heated to 200C. The sample is transferred to Module 3.

11.7.7.1.6. Module 3 is heated and the GC/MS column flow is routed through Module 3 to inject the sample and begin the run.

11.7.7.1.7. The system is pre-flushed with the next sample and the system is baked to limit carry-over.

11.7.7.2. For Cold Trap Dehydration technique, the autosampler will follow the sequence of events below (parameters may be modified based on instrument performance):

11.7.7.2.1. Blank (empty) trap (Module 1) is cooled to -40C and the Tenax trap (Module 2) is cooled to -40C.

11.7.7.2.2. Internal standard is trapped.

11.7.7.2.3. Sample is trapped.

11.7.7.2.4. Blank trap is heated to 10C. Any remaining sample is transferred to Module 2 by passing UHP Helium through Module 1. Conditions may vary based on instrument performance. This step is designed to remove water from the sample.

11.7.7.2.5. When GC is ready, the cryofocuser (Module 3) is cooled to –155C. Module 2 is heated to 200C. The sample is transferred to Module 3.

11.7.7.2.6. Module 3 is heated and the GC/MS column flow is routed through Module 3 to inject the sample and begin the run.

11.7.7.2.7. The system is pre-flushed with the next sample and the system is baked to limit carry-over.

11.7.8. Upon completion of the analytical sequence, the Entech software generates a QA/QC report that records data from the sampling event (i.e. actual volume trapped, temperature at the time of trapping, sample pressure, etc.).

11.8. Troubleshooting Guidelines

11.8.1. Many problems encountered during analysis are due to low standard pressures or carrier/detector gas supply issues. Always confirm that adequate pressure remains in the standards and that the instrument gas supplies are sufficient before working on the instrument hardware.

11.8.2. Low response – typically caused by leaking sample lines or valves or contaminated/dirty sources. Instrument software can perform automated leak checks of the system. Specific components can be checked by isolating the component in question from the system (disconnect and cap or plug the ends) and then performing a leak test using a pressure gauge and passivated canister at positive pressure. Leaking components will not hold pressure when the passivated canister is closed. Low internal standard areas may be caused by degradation of the MS performance and increasing the electron multiplier (EM) voltage may solve this concern.

11.8.3. Baseline noise – check for supply gas contamination and leaking fittings. Carrier gas filters may need to be changed, including the pencil filters inside the GC. Sample carry-over or contamination may also be an issue and baking the system while flushing sample lines will remove most carry-over. A dirty

source or leaking MS may also cause issues. The use of automated leak check routines in the MS software can indicate if a leak is present. Source-cleaning should be performed according to the manufacturer's instructions.

11.8.4. Tune issues – if an instrument will not pass tune the first step is to perform a mass axis calibration and peak-width adjustment. If the failure is due to ratios of ions with large differences, the tune parameters should be adjusted to achieve the desired ratios. The final corrective action is to clean the source according to the manufacturer's instructions.

11.8.5. Instrument issues – if data loss or error messages are encountered, consult the instrument troubleshooting guidance found in the operator's manual. The manual is in the help section of the GC software.

11.9. Maintenance or Repair of Analytical Instruments or Support Equipment

11.9.1. When analytical instruments or support equipment require repair or maintenance, they shall be taken out of operation or otherwise isolated, and tagged as 'out-of-service' until such a time as the repairs or maintenance have been made and the instrument or support equipment can be demonstrated as operational by calibration and/or verification or other tests to demonstrate acceptable performance. Details on the tag-out procedures to be followed may be found in the section of the QAM that discusses Equipment and Calibrations.

11.9.2. A new ICAL must be generated following major maintenance such as changing the column, cleaning or repairing the source, replacing filaments, changing electronics, replacing the multiplier or changing the concentrator.

11.9.3. Minor maintenance includes cleaning the injector port, replacing filters, changing the pump oil, autotuning, switching filaments (instrument contains two filaments under vacuum), replacing the syringe or injector tower, changing/refilling the calibration vial, changing seals and o-rings, ballasting pump, replacing fuses, replacing roughing pumps or transfer lines.

11.9.4. Schedule for routine maintenance of analytical instruments may be found in Attachment 10.

11.9.5. All maintenance or repair must be documented in the Instrument Maintenance Logbook.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Analyses

Two criteria must be satisfied to verify positive identification:

12.1.1. Elution of sample component at the same GC relative or absolute RT as those of the standard component.

12.1.1.1. The sample component relative retention time (RRT) must compare within ±0.06 RRT units of the RRT of the standard component.

12.1.1.2. As an option, RT must compare within 0.33 minutes of the standard component absolute RT. For reference, the RT standard must be run within the same 24-hour shift as the sample.

12.1.2. Correspondence of the sample component and the standard component mass spectra.

12.1.2.1. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.1.2.2. The relative intensities of ions must agree within ±30% between the standard reference and sample spectra. For example, for ions with ratio of 50% in the reference spectra, the corresponding sample ratio must be between 20 and 80%.

12.1.2.3. Standard reference mass spectra must be obtained on each individual GC/MS system.

12.1.3. If an analyte cannot be verified by all of the criteria in the above sections, but in the technical judgment of the analyst the identification is correct, then the analyte may be reported

12.1.3.1. Technical judgment may be based on whether a compound is present when co-elution occurs and a determination is made based on retention time and mass spectrum.

12.1.3.2. The difference in the spectra and the reason for the decision to report results must be explained in an NCM.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analysis being conducted. The following sections identify the guidelines for making tentative identification:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. Relative intensities of the major ions should agree within ±30%.

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.1.6. Only peaks having a total ion current greater than 10% of the nearest eluting IS total ion current will be evaluated for reporting.

12.2.2. TICs will be given general names consisting of major functional groups and number of carbon atoms unless an RT reference is available.

12.2.3. When TICs are requested to be reported using specific compound names, the following procedure must be followed:

12.2.3.1. Choose characterized ions of the specific compounds from the mass spectrum.

12.2.3.2. Search ions from expected RT range or entire RT range if the RT of the specific compound is unknown or uncertain.

12.2.4. Semi-quantitative results will be calculated for TICs using total ion current areas and assuming an RRF = 1.0.

12.2.5. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the analyst assign a tentative identification.

12.3. Quantitative Analysis

12.3.1. When an analyte has been identified, the quantification of that analyte will be based on the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. Quantitation will take place using

the IS technique.

12.3.2. A sample must be analyzed and reported at a dilution if one or more target analytes have an on-column amount above the upper calibration level. Dilutions are acceptable if at least one of the following criteria are met:

12.3.2.1. Any target analyte in the diluted sample is at or above the mid-point calibration standard (e.g., 8 ppbv on-column if 8 ppbv is the mid-point).

12.3.2.2. The peak height of any non-target analyte in the diluted sample exceeds the largest peak height of the highest calibration standard.

12.3.2.3. A heavy hydrocarbon matrix in the diluted sample raises the baseline two times that of the relative IS.

12.3.3. Analyte quantitation must be performed from the ICAL response and not from the CCV response. Test results must be qualified in reports when analyte quantitation is based on the CCV at client's request. This request must also be documented in an NCM and reported in the case narrative of the final report.

12.4. All manual or re-integration of chromatograms must be documented in accordance with TestAmerica Corporate SOP CA-Q-S-002. Documentation includes, at a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration, dated, and initialed. All manual integrations must undergo a secondary-level review.

12.5. Calculations

12.5.1. Calculation for RPD

$$RPD = \frac{Value\ A - Value\ B}{Average\ of\ Values} X\ 100$$

12.5.2. Calculation for RRF

$$RRF = \frac{Area\ cpd\ in\ Std.}{Area\ I.S.} X \frac{Conc.\ I.S.}{Conc.\ cpd\ in\ Std.}$$

The area of the primary quantitation ion is used in the calculation.

I.S. = Internal Standard

12.5.3. Calculation for %RSD

$$\%RSD = \frac{Std.\ Dev.\ of\ RRFs}{Mean\ of\ RRFs} X 100$$

12.5.4. Calculation for %D

$$\%D = \frac{Average\ RRF\ from\ ICAL - RRF\ CCV.}{Average\ RRF\ from\ ICAL} X100$$

12.5.5. Calculation for pressure DF

$$DF = \frac{Y_a}{X_a}$$

Where:

X_a = absolute canister pressure before dilution (initial pressure)
Y_a = absolute canister pressure after dilution (final pressure)

12.5.6. Calculation for Determining the Concentration of Compounds
The data system automatically quantitates the sample results based on a standard sample size of 250 mL. The default result units are in ppbv. If a sample size other than 250 mL was used or a canister sample was pressurized, the result must be adjusted as shown below:

$$Final\ result\ ppbv = raw\ result\ ppbv\ X \frac{250\ mL}{sample\ vol\ injected} X \frac{final\ psia}{initial\ psia}$$

Where:

$$rawresult\ ppbv = \frac{Area\ cpd\ in\ sample}{Area\ I.S.\ in\ sample} X \frac{Conc.I.S.}{RRF\ ICAL.}$$

Note: The area of the primary quantitation ion is used in the calculation.
I.S. = Internal Standard

12.5.7. Calculation for Percent Recovery (%Rec)

$$\%\,Rec = \frac{Amount\ cpd.\ recovered}{Amount\ cpd.\ spiked} X\ 100$$

12.5.8. Standard reporting units are ppbv (also ppb v/v). If results are to be reported in ng/L or ug/m^3, use the following equation:

$$result\ ppbv\ X \frac{Molecular\ weight\ of\ cpd}{24.45} = results\ ng/L\ or\ ug/m^3$$

Note: 24.45 is the molar volume of ideal gas in liters at 25°C and 1 atmosphere.

12.6. Estimates of uncertainty are based upon LCS historical control limits, and are provided on request only.

12.7. "J" values (results below the RL but above the MDL) are reported on request only.

12.8. No conversion of the analytical results to standard conditions is made.

12.9. The number of significant figures to be used when reporting sample and QC results are defined in QA Policy WS-PQA-004.

12.10. Technical Data Review

Technical data review is performed in accordance with Policy WS-PQA-012, and is documented utilizing review checklists. Examples of appropriate review checklists are found in Attachments 10 and 11.

12.10.1. One aspect of technical review is to ensure that the test instructions are clear, and that all project-specific requirements have been understood and followed. If directions to the analyst are not clear, the analyst must consult the Department Manager or the appropriate PM, who must clarify the instructions.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. This requires analysis of QC check samples containing all of the standard analytes for the method. It may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated

failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Expired standards will be part of the Lab Pack waste stream. They will be identified as expired, stored under the manufacturer's recommended conditions, and then packed for disposal as outlined in SOP WS-EHS-0001.

15.1.1. Gas standards in non-returnable, non-refillable cylinders, such as Scotty® Transportables, are slowly vented in the fume hood when empty. They are then turned over to the hazardous materials specialist, who ensures that they are damaged (e.g., a hole is drilled into the cylinders) so they cannot be reused. The damaged cylinders are then either recycled or scrapped.

15.1.2. Gas standards in returnable, refillable cylinders are returned to the manufacturer.

15.2. Air sample bags are slashed in a hood and then placed into an orange high VOA lab trash can. When the can is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 2nd edition, January 1999

16.1.1. Compendium Method TO-14A, Determination of Volatile Organic Compounds (VOCs) in Ambient Air using Specially Prepared Canisters With Subsequent Analysis Gas Chromatographic Analysis

16.1.2. Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)

16.2. TestAmerica Sacramento QAM, current revision

16.3. TestAmerica Sacramento SOP WS-QA-0032, Cleaning, Certification, and Preparation of Sampling Equipment, current revision

16.4. TestAmerica Corporate Environmental Health and Safety Manual CW-E-M-001, current revision

16.5. Advisory – Active Soil Gas Investigations, April 2012 (CAEPA, DTSC, LARWQCB, and SFRWQCB)

16.6. EPA/600/R-04/003, 2003 NELAC Standard, June 5, 2003

16.7. The NELAC Institute (TNI) Standard 2009, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis

16.8. TestAmerica Sacramento SOP WS-PQA-001, Quality Control Program, current revision

16.9. TestAmerica Corporate SOP CA-Q-S-005, Calibration Curves (General), current revision

16.10. TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices, current revision

16.11. TestAmerica Sacramento Policy WS-PQA-004, Rounding and Significant Figures, current revision

16.12. TestAmerica Corporate SOP CA-Q-S-006, Detection Limits, current revision

16.13. TestAmerica Sacramento SOP WS-EHS-0001, Sample and Chemical Waste Characterization, Collection, Storage and Disposal, current revision

17. METHOD MODIFICATIONS

17.1. Method TO-15 describes the use of zero air when making standards. TestAmerica Sacramento uses UHP nitrogen in the standards preparation.

17.2. UHP Nitrogen is used for dilution/pressurization purposes.

17.3. Method TO-15 describes canisters leak check being performed using pressure. TestAmerica performs the canisters leak check using vacuum unless specified by the project.

17.4. Method TO-14A describes an inlet system that uses a vacuum to pull the sample through the trap. TestAmerica Sacramento optionally uses the pressure of the sample canister to drive the sample through the trap.

17.5. Method TO-14A describes the use of a Nafion dryer to remove excess moisture from air matrices. TestAmerica Sacramento does not use a Nafion dryer since polar compounds may be lost during this removal step.

17.6. Method TO-14A describes the BFB tune check to be a gas sample introduced via a sample loop. TestAmerica Sacramento traps and analyzes BFB using the same analytical technique used with samples.

17.7. Methods TO-14A and TO-15 describe the use of passivated steel canisters for sampling and analysis. No mention is made of the use of air sample bags. TestAmerica Sacramento analyzes samples in air sample bags for VOCs using the same procedures described herein. A modification to the method is noted in the NCM submitted with the final report.

17.8. Methods TO-14A and TO-15 indicate that in order for the ICAL to be acceptable, all compounds must have a %RSD <30 (with allowance for two that could be up to 40% in TO-15). For routine analysis, for those analytes not listed in the published methods and are considered poor performers, TestAmerica Sacramento accepts the ICAL if up to four of these analytes have a %RSD ≤ 55. See Section 10.3.4. This modification accounts for analytical issues that arise from poor performing analytes.

17.9. For the continuing calibration criteria, Method TO-14A states that the RPD of each RF (in the CCV) from the mean RF of the ICAL curve should be <30%; for Method TO-15, the %D for each target compound must compare to the ICAL at ±30%. For routine analysis, for those analytes not listed in the published methods and are considered poor performers, TestAmerica Sacramento accepts the CCV if up to four of these analytes have a %D ±55. See Section 10.6.3. This modification accounts for analytical issues that arise for poor performing analytes.

17.10. Surrogates are not required by the methods. This SOP adds surrogates to every QC and client sample to help monitor for matrix effects and method performance. However, surrogates are not reported unless requested.

17.11. Method TO-15 states that the scan time must give 10 scans per peak, not to exceed 1 second per scan. The GC/MS software is set for a sampling rate of 3, which corresponds to approximately 2 to 3 scans per second, depending on the instrument.

See the GC/MS operator's manual or "help" on the software for more information about the sampling rate.

17.12. The transfer of sample from Tedlar bags to canisters is supported by the conclusions of an EPA poster titled LOSS/GAIN OF VOCS FROM TEDLAR BAGS AND OTHER SAMPLING EQUIPMENT, by C. Loss Paul as presented at the Presented at The 17th Annual Association for Environmental Health and Sciences Meeting, San Diego, CA, March 21 - 22, 2007. According to the synopsis,

> Soil gas samples are collected to evaluate human health risk from vapor intrusion into homes and other buildings. In order to meet risk assessment goals, the analytical reporting limit for many compounds of concern are down to part per billion ranges. The appropriate sampling tubing, sample containers and leak check compounds should be selected in order to ensure data quality objectives. Mechanisms which can impact sample integrity include adsorption of volatile organic compounds (VOCs) onto the sampling media and diffusion of VOCs through the sampling media which may result in artificially low values, and desorption of compounds from the sampling media into the sample which may results in artificially high values. A literature search was conducted to compile published results of impacts on soil gas samples from sampling equipment (i.e., tubing) and containers (i.e., Tedlar® bags). The literature search revealed that the recommended holding time for samples stored in Tedlar® bags not exceed 48 hours. A laboratory study was conducted to evaluate the holding times for certain VOCs in 1 L Tedlar® bags stored at two different temperatures. VOCs used in this experiment include 1,1,1-trichloroethane (1,1,1-TCA), trichloroethylene (TCE), benzene, and toluene that were combined in a gas mixture with nitrogen. Bags were filled with the gas mixture and then stored for different times and temperatures. Two incubators were set at two temperatures, C to simulate field temperatures and the bags were stored from 8°C and 25°15 hours to 2 weeks. Results of this study show that 1,1,1-TCA, TCE, and benzene can be stored up to one week without significant impact on concentrations. Results for toluene are less conclusive. However, soil gas samples collected in Tedlar® bags should be analyzed as quickly as practical or samples can be transferred to another container with longer holding times (i.e., Summa canister).

18. ATTACHMENTS

18.1. Attachment 1: Standard Analytes, Reporting Limits, and Characteristic Ions

18.2. Attachment 2: BFB GC Operating Conditions, EPA TO-15

18.3. Attachment 3: GC Operating Conditions, EPA TO-15

18.4. Attachment 4: BFB Acceptance Criteria, EPA TO-14A

18.5. Attachment 5: BFB Acceptance Criteria, EPA TO-15

18.6. Attachment 6: Internal Standards

18.7. Attachment 7: Surrogate Standards

18.8. Attachment 8: Example GC/MS Initial Calibration Curve Review Checklist

18.9. Attachment 9: Example GC/MS Technical Data Review Checklist

18.10. Attachment 10: Schedule for Routine Maintenance of Analytical Instrument

18.11. Attachment 11: Canister Pressurization Logbook (Example Page)

18.12. Attachment 12: GRO Analysis

19. REVISION HISTORY

19.1. WS-MSA-0015, Revision 1.3, Effective 08/15/2014

19.1.1. Section 8, revised second row of table to reflect the more recent "Advisory, Active Soil Gas Investigations" (from 2012 rather than 2003). Reference in 16.5 also changed.

19.1.2. Inserted Section 5.1.2, safety requirements when transferring sample from air bags into syringes or canisters.

19.1.3. Section 8 – in accordance with the most recent DTSC advisory, changed the holding time to 30 days for canisters under DTCS requirements as well.

19.1.4. Changed Section 7.2.2 from 'concentration of 250 ppbv…' to 'concentration of 300 ppbv…'.

19.1.5. Changed Section 7.3.1.1 from 125 µL to 50 µL Distilled or Nanopure water.

19.1.6. Inserted Section 9.7.1, referring to DoD requirements.

19.1.7. Changed Section 10.2.7 to – "The nominal concentrations of the ICAL standards are typically 0.30, 0.40, 0.80, 2.0, 4.0, 8.0, 20, 40 and 60 ppbv, but these may vary depending on the certified mix used to prepare the standards or the volume trapped. The low standard must be at or below the RL. The standards are analyzed by preparing stock standards at the required concentration or by varying the trapped volume of the working standards from the default volume of 250 mL. For example, the 0.30, 0.40, and 0.80 ppbv standards are analyzed by trapping 30, 40, and 80 mL, respectively, of a 2.5 ppbv working standard."

19.1.8. Removed Section 11.2.4, 11.2.4.1, and 11.2.4.2, which discussed the laboratory default to analyze air sample bags at a 20x dilution.

19.1.9. Removed the subheadings from Section 11.4 (Screening).

19.1.10. Section 11.7.2, appended the following, "The sequence is verified by another analyst. This analyst verifies the autosampler sequence, port position, chem. station sequence, and Chrom worklist. The analyst annotates in the instrument maintenance log if the sequence has been verified."

19.1.11. Changed Section 11.7.4 from – 'If the default volume is 250 mL then 410 mL should be trapped' to 'If the default volume is 250 mL then 510 mL should be trapped.'

19.1.12. Combined GC operating conditions and BFB operating conditions (Attachment 2 and Attachment 3) as they are the same. Renumbered the attachments.

19.1.13. Updated Attachments 7, 8, and 10 to current versions.

19.1.14. Editorial comments.

19.1.15. Inserted Attachment 11 – GRO Analysis using Method TO-15

19.1.16. Editorial changes.

19.2. WS-MSA-0015, Revision 1.2, Effective 10/25/2013

19.2.1. Added psig to psia conversion in section 3.10.

19.2.2. Removed reference to Costa Mesa SOP LA-QAS-002.

19.2.3. Added language to clarify "certified clean" in section 8.2.

19.2.4. Changed humidification of method blank canister from 100uL to 50uL with distilled or NanoPure water in section 9.3.

19.2.5. Added language to evaluate EICP for S/N determination in section 10.4.1.

19.2.6. Insert section 10.4.2 to state qualitative compound identification criteria must be met for all concentrations.

19.2.7. Insert 10.4.5 to add language for re-fit / read-back evaluation for % error.

19.2.8. Removed section 11.5 from previous version regarding water addition.

19.2.9. Added language in section 11.7.6 for sample dilution that can is pressurized to 24-26 psia or no more than 3x the initial pressure.

19.2.10. Added language in method modification to use UHP Nitrogen for standards preparation instead of zero air, section 17.1.

19.2.11. Added language in method modification that UHP Nitrogen is used for dilution / pressurization, section 17.1

19.2.12. Added language in method modification that leak check is performed by measuring vacuum instead of pressure as stated in the source method, section 17.3.

19.3. WS-MSA-0015, Revision 1.1, Effective 04/22/2013

19.3.1. Inserted Section 5.1 to address transfer of sample from Tedlar bags to canisters.

19.3.2. Added Section 17.11 regarding the supporting evidence for transferring samples from Tedlar bags to canisters to prolong the holding time.

19.3.3. Updated Attachment 1 to reflect the analytes evaluated at this time.

19.3.4. Removed Attachment 2 until such time as an additional mix is introduced to the analysis.

19.3.5. Section 9.2, removed references to LCS Duplicate.

19.3.6. Inserted Section 9.1.1 regarding LCS duplicates.

19.3.7. Removed all other references to LCS duplicates (aside from Section 9.1.1)

19.3.8. Replaced all references to LA-SRA-002 with WS-QA-0032.

19.4. WS-MSA-0015, Revision 1, Effective 04/08/2013

19.4.1. Revised Attachment 3 to update conditions.

19.4.2. Revised Attachment 4 to update conditions.

19.4.3. Editorial revisions.

19.5. WS-MSA-0015, Revision 0, Effective 01/18/2013

19.5.1. This is the first version of this SOP.

Attachment 1: Standard Mix Analytes and Characteristic Ions

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
1,1,1,2-Tetrafluoroethane	811-97-2	86	69, 51	1.0
1,1,1-Trichloroethane	71-55-6	97	99, 61	0.30
1,1,2,2-Tetrachloroethane	79-34-5	83	85, 131	0.40
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	101	151, 103	0.40
1,1,2-Trichloroethane	79-00-5	97	83, 85	0.40
1,1-Dichloro-1-fluuoroethane	1717-00-6	81	61, 45	0.40
1,1-Dichloroethane	75-34-3	63	65, 83	0.30
1,1-Dichloroethene	75-35-4	61	96, 98	0.80
1,1-Dichloropropene	563-58-6	75	110, 39	0.40
1,1-Difluoroethane	75-37-6	51	65	0.40
1,2,3-Trichlorobenzene	87-61-6	180	182, 145	2.0
1,2,3-Trichloropropane	96-18-4	110	97, 75	0.40
1,2,4-Trichlorobenzene	120-82-1	180	182, 145	2.0
1,2,4-Trimethylbenzene	95-63-6	120	105, 77	0.80
1,2-Dibromo-3-chloropropane	90-12-8	157	155, 75	2.0
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	135	85, 87	0.40
1,2-Dichlorobenzene	95-50-1	146	148, 111	0.40
1,2-Dichloroethane	107-06-2	62	49, 64	0.80
1,2-Dichloropropane	78-87-5	76	63, 62	0.40
1,3,5-Trimethylbenzene	108-67-8	120	105, 77	0.40
1,3-Dichlorobenzene	541-73-1	146	148, 111	0.80
1,3-Dichloropropane	142-28-9	76	41,78	0.80
1,4-Dichlorobenzene	106-46-7	146	148, 111	0.40
1,4-Difluorobenzene	540-36-3	114	88, 63	
1,4-Dioxane	123-91-1	88	58	0.80
1.1.1.2-Tetrachloroehtane	630-20-6	131	133, 95	0.40
2,2-Dichloropropane	78-87-5	77	97, 41	0.80
2-Butanone (MEK)	78-93-3	72	43, 57	0.80
2-Chlorotoluene	95-49-8	126	91, 89	0.40
2-Hexanone	591-78-6	58	43, 85100	0.40
2-Methyl-2-propanol	75-65-0	59	57, 41	2.0
3-Chloro-1-propene (Allyl Chloride)	107-05-1	41	76, 78	0.80
4-Ethyltoluene	622-96-8	120	105, 77	0.40
4-Isopropyltoluene	99-87-6	119	134, 91	0.80
4-Methyl-2-pentanone (MIBK)	108-10-1	43	58, 100	0.40
Acetone	67-64-1	43	58	5.0
Acrolein	107-02-8	56	55, 53	2.0
Alpha Methyl Styrene	98-83-9	118	103, 117	0.40
Benzene	71-43-2	78	77, 52	0.40

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
Benzyl chloride	100-44-7	91	126, 63	0.80
Bromobenzene	108-86-1	156	77, 158	0.80
Bromoform	75-25-2	173	175, 171	0.40
Bromomethane	74-83-9	94	96, 79	0.80
Butadiene (1,3-Butadiene)	106-99-0	39	54, 53	0.80
Butane	106-97-8	43	41, 58	0.40
Carbon disulfide	75-15-0	76	78, 44	0.80
Carbon tetrachloride	56-23-5	117	119, 121	0.80
Chlorobenzene	108-90-7	112	114, 77	0.30
Chlorodibromomethane	124-48-1	129	127, 131	0.40
Chlorodifluoromethane	75-45-6	51	67, 50	0.80
Chloroethane	75-00-3	64	49, 66	0.80
Chloroform	67-66-3	83	85, 47	0.30
Chloromethane	74-87-3	50	52	0.80
cis-1,2-Dichloroethene	156-59-2	96	61, 98	0.40
cis-1,3-Dichloropropene	10061-01-5	75	77, 39	0.40
Cyclohexane	110-82-7	84	56, 69	0.40
Cyclohexanone	108-94-1	55	42, 98	0.80
Dibromomethane	74-95-3	174	93, 95	0.40
Dichlorobromomethane	75-27-4	85	83, 129	0.30
Dichlorodifluoromethane	75-71-8	85	87, 50	0.40
Dichlorofluoromethane	75-43-4	67	69	0.40
Ethanol	64-17-5	46	45	1.0
Ethyl acetate	141-78-6	43	61, 70	0.30
Ethylbenzene	100-41-4	91	106, 65	0.40
Ethylene Dibromide (EDB)	106-93-4	107	109, 81	0.80
Hexachlorobutadiene	87-68-3	225	223, 227	2.0
Hexane	110-54-3	43	57, 86	0.80
Iodomethane	74-88-4	142	127	0.40
Isooctane (2,2,4-Trimethylpentane)	540-84-1	57	41, 56	0.40
Isopropyl alcohol	67-63-0	45	4359	0.80
Isopropyl ether	108-20-3	45	87, 59	0.80
Isopropylbenzene	98-82-8	120	105, 79	0.80
Methyl methacrylate	80-62-6	69	41, 100	0.80
Methyl tert-butyl ether	1634-04-4	73	57, 41	0.80
Methylene Chloride	75-09-2	49	84, 86	0.40
m-Xylene & p-Xylene	179601-23-1	91	106, 77	0.80
Naphthalene	91-20-3	128	102, 129	0.80
n-Butanol	71-36-3	56	41, 43	2.0
n-Butylbenzene	104-51-8	92	91, 134	0.40
n-Heptane	142-82-5	43	41, 100	0.80

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
n-Nonane	111-84-2	43	57, 85	0.80
n-Octane	111-65-9	43	41, 85	0.40
N-Propylbenzene	103-65-1	91	120, 65	0.40
o-Xylene	95-47-6	91	106, 105, 78	0.40
Pentane	109-66-0	43	57, 72	0.80
Propane	74-98-6	43	42, 39	0.40
Propane, 2-methyl	75-28-5	43	41	0.40
Propene	115-07-1	41	39, 42	0.40
sec-Butylbenzene	135-98-8	105	134, 91	0.40
Styrene	100-42-5	104	78, 103	0.40
Tert-amyl methyl ether (TAME)	994-05-8	73	55, 87	0.80
Tert-butyl ethyl ether	637-92-3	59	87, 57	0.80
tert-Butylbenzene	98-06-6	91	119, 134	0.80
Tetrachloroethene	127-18-4	166	164, 131	0.40
Tetrahydrofuran	109-99-9	42	71, 72	0.80
Toluene	108-88-3	91	92, 65	0.40
trans-1,2-Dichloroethene	156-60-5	61	96, 98	0.40
trans-1,3-Dichloropropene	10061-02-6	75	77, 39	0.40
Trichloroethene	79-01-6	130	95, 132	0.40
Trichlorofluoromethane	75-69-4	101	103, 66	0.40
Vinyl acetate	108-05-4	43	86, 42	0.80
Vinyl bromide	593-60-2	106	108, 81	0.80
Vinyl chloride	75-01-4	62	64, 61	0.40
Xylenes, Total	1330-20-7	NA	NA	1.20
Internal Standards				
Chlorobromoethane	107-04-0	130	49.128	
1,4-Difluoroethane	540-36-3	114	88, 63	
Chlorobenzene-d5	3114-55-4	117	82. 54	
Surrogates				
1,2-Dichloroethane-d4	3855-82-4	85	87, 102	
Toluene-d8	2037-26-5	100	98	
4-Bromofluorobenzene	460-00-4	95	174, 176	

Attachment 2: GC Operating Conditions, BFB and EPA TO-15

Method file: TO15				
Method File List				
GC Type: 6890				
Run type: Scan, GC, E1				
Column: Cap				
Split Ratio: 40.5:1				
	Inj.P	**Intfc**	**Source**	
Temp, °C	**150°C**	**220°C**	**NA**	
GC/DIP				
Initial Column Temperature / Hold time	**35°C for 5 minutes**			
Temperature Profile	**35°C - 110°C at 6.0°C /minute, hold for 0.10 minute, 110°C - 220°C at 8.0°C, hold for 1 minute**			
Post Time	**5.0 minutes**			
Oven equilibration Time: 0 min				
Run time: 37.35 min				
Scan Start time: 3.95 min				
Scan Parameters:				
Mass Range: 35 to 300				
Multiplier voltage: varies				
Number of samples: 2				
Threshold: 200 counts				

Attachment 3: BFB Acceptance Criteria, EPA TO-14A

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2.0% of mass 174
174	>50% of mass 95
175	5.0 to 9.0% of mass 174
176	>95% but <101% of mass 174
177	5.0 to 9.0% of mass 176

Attachment 4: BFB Acceptance Criteria, EPA TO-15

Mass	Ion Abundance Criteria
50	8.0 to 40% of mass 95
75	30 to 66% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2% of mass 174
174	50 to 120% of mass 95
175	4.0 to 9.0% of mass 174
176	93% to 101% of mass 174
177	5.0 to 9.0% of mass 176

Attachment 5: Internal Standards

Bromochloromethane
1,4-Difluorobenzene
Chlorobenzene-d5

Attachment 6: Surrogate Standards

1,2-Dichloroethane-d4
Toluene-d8
4-Bromofluorobenzene

Attachment 7: Example GC/MS Initial Calibration Curve Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

ICAL Review Checklist
Volatile Air GCMS

Method & Instrument ID: _____ **ICAL Date:**_____
Chrom Worklist #:_____ **Calibration ID:** _____ **TALS Analysis Batch:** _____
Method (Check the applicable box)
☐ TO-14A / TO-15 Modified SCAN ☐ TO-15 Scan ☐ TO-15 SIM ☐ TO-15 SIM (Special Project)

Acceptance criteria are found in the applicable laboratory SOP or in TALS. If item is No, create an NCM.

Review Items	Level 1		NA	Level 2
General Criteria	Yes	No		
Standards analyzed within 24 hour tune time.				
Chrom Peak Review calibration reports (F6) reviewed for error flags				
Retention time correct for isomers/coeluters and all other analytes • Dichlorodifluoromethane / 1,2-Ddichlorofluoroethane • Trichlorofluoromethane / 1,1,2-Trichlorofluoroethane • Hexane / Vinyl acetate • 2-Methyl butane / Acrolein • 1,1-Dichloroethene / cis-1,2-Dichloroethene / trans-1,2-Dichloroethene • 1,2-Dichloroethane / Benzene • cis-1,3-Dichloropropane / trans-1,3-Dichloroproapne • Ethylbenzene / m/p-Xylene / o-Xylene • 4-Ethyl toluene / 1,3,5-Trimethylbenzene / 1,2,4-Trimethylbenzene • tert-Butylbenzene / 4-Isopropyltoluene • 1,3-Dichlorobenzene / 1,4-Dichlrobenzene / 1,2-Dichlorobenzene • 1,2,4,5-tetramethylbenzne / 1,2,3,4-tetramethylbenzene • 1,2,4-Trichlorobenzene / 1,2,3-Trichlorobenzene				
Curve reviewed in Chrom for all analytes and all appropriate leves are included/excluded				
Uploaded all calibration levels to TALS after any changes were made in Peak Review				
Method for this worklist set as Most Recent Method				
ICAL locked in Chrom and in TALS				
ICV passes -See criteria below				
Manual Integrations clearly identified, initialed, dated and reason given.				
SIM Criteria				
PFTBA tune documentation present in Chrom & TALS and meets criteria				
RSD < 30% (≤ 40% for up to 2 analytes.				
Linear regressions >0.995 and intercept <+/- ½ RL/IS amount, (minimum of 5 points curve).				
All internal standards are within 60-140% of ICAL midpoint.				
ICV (Second Source) meets 70-130% criteria (60-140%) for up to 2 analytes,				
Scan Criteria				
BFB Tune documentation present in Chrom & TALS and meets criteria				
For GRO: WS-GRO report (F8) reviewed to ensure IS/Surrogate subtraction from area sum.				
RSD < 30% (≤ 40% for up to 2 analytes, and ≤ 55% for up to 4 poor performers defined in SOP)				
Linear regressions >0.995 and intercept <+/- ½ RL/IS amount, (minimum of 6 points curve).				
All internal standards are within 60-140% of ICAL midpoint (for TO-15 / TO-15 Modified) or within 50-200% of ICAL midpoint (for TO-14A)				
ICV (Second Source) meets 70-130% criteria (60-140% for up to 2 analytes, and 45-155% for up to 4 poor performers defined in SOP)				

	Repeat Failure from Prev ICAL Yes	No
ICAL compounds outside RSD criteria (write compound and %RSD _____		
ICV (second source) compounds outside RSD criteria (write compound and %D. _____		

1st Level Reviewer: _____Date: _____

2nd Level Reviewer: _____Date: _____

Comments: _____

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Attachment 8: Example GC/MS Technical Data Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

MS VOA AIR Data Review Checklist

Job(s):_____ Instrument ID: _____

☐TO-14A ☐TO-14A MOD ☐TO-15 ☐TO-15 MOD ☐TO-15 SIM

ICAL Batch: _____ TALS Batch:_____

Review Items	Level 1 Yes	Level 1 No	Level 2	NA
Initial Calibration				
1. Is ICAL locked in Chrom & TALS?				
2. Is ICV properly linked in TALS for L3/L4 jobs?				
Continuing Calibration				
1. BFB tune documentation meets criteria? The PFTBA Tune Documentation is attached in TALS (SIM methods only)?				
2. All Internal Standards within 50-200% of ICAL mid-point (TO-14A/TO-14A MOD) or 60-140% (TO-15/ TO-15 MOD?				
3. Does the %D meet:30%D for standard program, and 20%D for DoD?				
4. Isomeric pairs checked for correct peak assignment? • Dichlorodifluoromethane / 1,2-Ddichlorofluoroethane • Trichlorofluoromethane / 1,1,2-Trichlorofluoroethane • 2-Methyl butane / Acrolein • 1,1-Dichloroethene / cis-1,2-Dichloroethene / trans-1,2-Dichloroethene • Hexane / Vinyl acetate • 1,2-Dichloroethane / Benzene • cis-1,3-Dichloropropane / trans-1,3-Dichloroproapne • Ethylbenzene / m/p-Xylene / o-Xylene • 4-Ethyl toluene / 1,3,5-Trimethylbenzene / 1,2,4-Trimethylbenzene • tert-Butylbenzene / 4-Isopropyltoluene • 1,3-Dichlorobenzene / 1,4-Dichlorobenzene / 1,2-Dichlorobenzene • 1,2,4,5-tetramethylbenzne / 1,2,3,4-tetramethylbenzene • 1,2,4-Trichlorobenzene / 1,2,3-Trichlorobenzene				
Client Samples & QC Sample Results				
1. Was analysis done within holding times?				
2. Are Chromatograms reviewed and spectra verified?				
3. Are positive results within calibration range?				
4. Are all positive results within RT windows?				
5. All target cpds in MB < RL (< ½ RL for DoD)? Requires NCM if "no."				
6. Are surrogate recoveries for MB and LCS/LCSD within control limits?				
7. Are target constituents in LCS/LCSD within control limits?				
8. Are all Chrom graphics uploaded?				
9. Are all QC samples properly linked in TALS?				
10. Do the Internal Standards meet criteria +/-50% to +/-200% (TO-14A / TO-14A MOD), +/-60% to +/- 140% (TO-15/TO-15 MOD)?				
11. Do results (e.g., dilutions) make sense?				
12. Are MS/MSD recoveries and RPDs within control limits?				
13. Have all flags been reviewed for appropriateness?				
14. All manual integrations appropriate and documented?				
15. Are non-conformances documented as NCMs?				
16. Are client sample surrogate failures flagged and NCM written?				
17. Dilutions due to target cpds? ____ Dilutions due to non-targets? ____				
18. Has QC checker been run?				

1st Level Reviewer / Date:_____ 2nd Level Reviewer / Date: _____

Comments: _____

q:\forms\checklists\msa-002 air gcms data review 2014-02-24.doc MSA-002 RKE 07/16/2014

Attachment 9: Schedule for Routine Maintenance of Analytical Instrument

Frequency	Maintenance Item
Daily	Check baseline level with analysis of blanks. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. Autosampler: Check for proper operation. Leak check system.
As needed	Replace septum. Clean injector port. Break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add or change oil, if needed. Replace the exhaust filters on the mechanical rough pump every 1-3 years, or as needed. Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels. Check ion source and analyzer (clean, replace parts as needed). Clean source, including all ceramics and lenses - as is indicated by a variety of symptoms including inability to properly tune, poor response, and high background contamination. Repair/replace jet separator. Replace filaments when burnt out or performance indicates need for replacement.

Attachment 10: Canister Pressurization Logbook (Example Page)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

Canister Pressurization Log

Date	Sample ID	Can #	Initial Press. (psig)	Initial Press. (psia)	Final Press. (psig)	Final Press. (psia)	Gauge ID	Initial	Comments
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		

A = Gauge CD5437; **B** = Gauge _____

[Calculation: psig + 14.7 = psia]

Reviewed by /Date: _____

QA-813 r1 GEC 7/21/2014

PAGE 1

1. SCOPE AND APPLICATTION

1.1. This method describes the analysis of volatile petroleum hydrocarbons. The procedure used for the petroleum hydrocarbons is based on SW-846. Refer to Table A1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 218°C. Classes of compounds best suited for evaluation by this analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

2. SUMMARY OF METHOD

Refer to Section 2 of the main body of this SOP for a summary of the method.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of the method.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for Safety Information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for equipment and supplies.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP Helium for carrier gas

7.1.2. UHP H2 for detector combustion

7.1.3. Zero-grade air for detector combustion

7.1.4. UHP N2 for sample preparation, standard preparation, and method blanks

7.1.5. Liquid N2

7.2. Standards – Neat standards are good for 1 year from date purchased or if a vendor supplied standard has an earlier expiration date then that date is used.

7.2.1. Certified BTEX, MTBE, and alkane standards in pressurized gas cylinders and neat liquids, obtained from select NIST-approved vendors.

7.2.2. Certified Hexane standards in pressurized gas cylinder and neat liquid, obtained from select NIST-approved vendors.

7.2.3. Certified neat unleaded gasoline standards, obtained from select NIST-approved vendors.

7.3. Standard Preparation

Standard preparation activities are performed accordance with TestAmerica SOP WS-QA-0017.

7.3.1. Dilutions of standards are made on a volume/volume basis using serial dilution methodologies. UHP N_2 is used as the diluent gas.

7.3.2. Primary gas standards are either purchased from manufacturers in pressurized, pre-mixed gas cylinders, or prepared in passivated canisters or air sample bags from neat liquid standards.

7.3.2.1. Primary Gas Standard Prepared in Canisters - To prepare a primary gas standard from neat liquids, an aliquot of the compound is injected into a clean and evacuated (-30 inches of mercury) passivated canister. The density of the compound is used to determine the mass injected. The canister is pressurized using UHP N2 and is then allowed to equilibrate.

7.3.2.2. Primary Gas Standard Prepared in Air Sample Bags - To prepare a primary gas standard from neat liquids, an aliquot of the compound is injected with a volumetric gas-tight syringe into a new and clean air sample bag. The air sample bag is filled with UHP N2 to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

To calculate the concentration of the standard prepared:

$$ppmv = \frac{\left(D \times V_1 \times 24.45 \times 10^6\right)}{\left(MW \times V_2\right)}$$

Where:

V_1 = aliquot of neat liquid (mL)

D = density of neat liquid (g/mL)

V_2 = final volume in passivated canister or air sample bag (L)

24.45 = molar volume (L/mole) at STP

10^6 = conversion factor

MW = molecular weight (g/mole)

7.3.3. Working gas standards are good for 6 months from date of preparation. They are prepared in passivated canisters or air sample bags by making dilutions of the primary pre-mixed gas cylinders (from vendors) or by making dilutions of the prepared primary gas standard prepared from the neat liquid standards (see Section 7.3.2).

7.3.3.1. Working Gas Standard Prepared in Canisters - To prepare a working gas standard, an aliquot of the prepared primary gas standard or of the pre-mixed primary gas standard is transferred to a clean and evacuated passivated canister. The aliquot is metered in by measuring the vacuum of the canister as the aliquot is being transferred. The ratio of the final canister pressure and the pressure transferred is the dilution factor. The canister is pressurized with UHP N2 and is then allowed to equilibrate.

7.3.3.2. Working Gas Standard Prepared in Air Sample Bags - To prepare a working gas standard, an aliquot of the prepared primary gas standard or of the pre-mixed primary gas standard is transferred to a new and clean air sample bag. The aliquot is transferred by direct injection with a volumetric gas-tight syringe. The air sample bag is filled with UHP N2 to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

7.3.3.3. To calculate the concentration of the standard prepared:

$$ppmv = C_{ps} \times {P_i}/{P_f}$$

Where:

C_{ps} = concentration of compound in primary gas standard, ppmv

P_i = aliquot of primary gas standard used in psia or in mL if using air sample bag

P_f = final pressure of canister in psia or final volume in mL if using air sample bag

7.3.4. Gasoline Calibration Standards

7.3.4.1. The primary standards are prepared in passivated canisters from neat unleaded gasoline. Gasoline from one vendor will be used to prepare two or more gasoline calibration standards. The second

source LCS is prepared from unleaded gasoline obtained from a different lot number.

7.3.4.2. A 10-, 25-, 50-, or 100-µL gas-tight syringe is used to transfer the neat gasoline into the septum-capped evacuated canister. The total (barrel + needle) syringe volume is accounted for.

8. SAMPLE PRESERVATION AND STORAGE

Refer to Section 8 of the main body of this SOP for information regarding sample preservation and storage.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the main body of this SOP for general quality control requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the % RSD of the response factor or calibration factor of each analyte is ≤ 30%. If an analyte exceeds the criteria, then a regression line (linear or curved) may be attempted.

10.1.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the **normal** concentration/response behavior of the detector.

10.1.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is ≤ 30%. The equation for average response factor is:

Equation A1

$$\textit{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.1.3. Linear regression

The linear fit uses the following function:

Equation A2

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response

x = Concentration

a = Slope

b = Intercept

10.1.4. Quadratic curve

The quadratic curve uses the following function:

Equation A3

$$x = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where: x = concentration

y = instrument response

a = 2nd order coefficient (curvature)

b = 1st order coefficient (slope)

c = constant (intercept)

10.2. Evaluation of calibration curves

The %RSD from the calibration curve is used to evaluate the initial calibration. If a regression line is used, the coefficient of determination (r2) shall be greater than or equal to 0.990, or the correlation coefficient (r) shall be greater than or equal to 0.995, whichever is appropriate to the regression fit used.

10.3. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response should be less than ± ½ the reporting limit for the analyte.
- The RSD for average response factors for each component must be ≤ 20%.
- When RSD is inappropriate, either the correlation coefficient (r) must be ≥ 0.995, or the coefficient of determination (r^2) must be ≥ 0.990.

10.4. Initial Calibration (ICAL)

10.4.1. At the beginning of each initial calibration curve for gasoline or other hydrocarbon fuel mixtures, the carbon range is established from the n-alkanes in the calibration standard. The start and end points for the peak summing range is the retention time at the apex of the beginning and ending carbon

range of the fuel mixture (e.g RT of C6 to RT of C12 measured at the beginning of the start peak and the end of the end peak.

10.4.2. The initial calibration for the GRO curve consists of 6 calibration standards (concentrations listed in Table A3)

10.5. Quantitating hydrocarbon mixtures:

Starting and ending retention times for quantitation are determined for each fuel as per 10.4.1. The peak areas between the starting and ending times are summed and 1,4-Difluorobenzene is used to generate a response factor. This factor is used to quantitate sample results, and depending on the client requirements, this factor may be applied to the same retention range as the standard, or to a different range.

10.6. Integration

10.6.1. When evaluating the initial calibration, also evaluate hydrocarbon pattern integration.

10.6.1.1. The integrated peaks should run from the point in the chromatogram prior to peaks to a region following the mixture's peaks in a fairly straight line.

10.6.2. The default integration parameters generated as a result of evaluating the initial calibration should remain in effect until the initial calibration is reanalyzed.

10.7. Evaluate the initial calibration as per 10.1 and 10.3.

10.8. A second source standard is analyzed immediately following the initial calibration. The acceptance criteria for the second source standard is ± 30% from the expected value.

10.9. Continuing Calibration Verification (CCV)

10.9.1. A CCV (5000ppbv or another mid-level standard) is analyzed at the beginning of each 24 hour analytical sequence. The integration for the GRO CCV follows the same technique as stated in 10.6.

10.9.2. The acceptance criteria for the CCV is ± 30% D.

11. PROCEDURE

11.1. Refer to Section 11 of the main body of this SOP for information regarding sample preparation and other procedures.

12. CALCULATIONS/DATA REDUCTION

12.1. Refer to Section 12 of the main body of this SOP for general information regarding data analysis and calculations.

12.2. Gasoline is quantitated using the area between two marker peaks as specified in Table A2.

12.3. Calculate results in samples as in Section 12 of the main portion of this SOP.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance criteria.

14. POLLUTION CONTROL

Refer to Section 14 of the main body of this SOP for information relating to pollution control.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for information relating to waste management.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035, 8000B, and 8015B.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, March 2003, Section 8000C.

16.3. Leaking Underground Fuel Tank Field Manual, State of California, October, 1989

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. This method is performed on a GC/MS, with surrogate values calculated using Method TO-14A/TO-15, and the TPH values calculated using the total area chromatogram. All of the reference methods are based on GC-FID as the detector and the limitations thereof.

18. ATTACHMENTS

18.1. Table A1 – Standard Analyte List

18.2. Table A2 – Volatile Petroleum Hydrocarbon Quantitation Ranges.

18.3. Table A3 - Calibration Standard Concentrations

18.4. Table A4 - LCS Spiking

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Table A1		
Standard Analyte List		
Compound	CAS Number	Reporting Limit (ppbv/v)
Gasoline Range Organics (GRO)	NA	100

Table A2		
Volatile Petroleum Hydrocarbon Quantitation Ranges.		
Regulatory Method GRO	Quantitation Range	
	Start	End
GRO	n-C3	n-C12
nC4-nC12	nC4	nC12
C4-C12	n-C4	n-C12

[1] *The method specifies that the peak summing window must start at the start of the n-C6 peak and finish at the start of the n-C10 peak.*

Table A3						
Calibration Standard Concentrations (ppbv/v)						
Component	1	2	3	4	5	6
Gasoline	200	500	1000[1]	5,000	10,000	20,000

[1]*Designates the mid-level standard concentration used for the ICV and CCVs following the initial CCV of a sequence.*

Refer to the main body of this SOP for surrogate details.

Table A4	
LCS Spiking	
Component	Concentration (ppbv)
GRO	5000

Title: Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry [Methods 200.8, 6020 and 6020A]

Approvals (Signature/Date):

Thep Phomsopha — 12/3/14 — Date Technical Manager	Joe Schairer — 12/5/14 — Date Health & Safety Manager / Coordinator
Lisa Stafford — 12/4/2014 — Date Quality Assurance Manager	Robert Hrabak — 12/4/14 — Date Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA Method 200.8, 6020 and 6020A.

1.2. Table 1 lists the analytes contained in the standard analyte list for ICPMS. These are the analytes for which the laboratory maintains certification in the form of PT samples, MDLs, IDLs, DOCs and linear range analyses. Elements not in this list may be analyzed with the client's knowledge that we do not maintain certification for analytes, only standards, outside the scope of our standard product.

1.3. Methods 200.8, 6020 and 6020A are applicable to the determination of total recoverable analyses in ground water, surface waters, and TCLP, STLC, and SPLP leachates/extracts. These methods are also applicable to the total analysis of soils, sludges, sediments and wastes. All matrices require digestion prior to analysis with the exception of STLC extracts. At a minimum, dissolved samples and STLCs are matrix matched under the guidelines of WS-IP-0008. STLCs are to be matrix matched at a 10x dilution. Silver concentrations must be below 0.1 mg/L in aqueous samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data

1.4. Reporting Limits

1.4.1. The standard reporting limits for metals analyzed by ICP-MS are listed in Table 1.

1.4.2. Reporting limits may differ from those listed if prescribed in a reviewed and finalized client specific QAPP.

1.4.3. Reporting limits for samples analyzed under QSM and AFCEE criteria usually differ from those shown in Table 1 and in many cases are specific to the client.

1.5. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates or leachates are introduced via a cross flow nebulizer into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio-frequency plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber

that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Isobaric interferences: Isobaric interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.

4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences.

4.3. See Tables 8 and 9 for a list of common interferences and the isotopes affected.
Note: Not all-possible interferences are listed.

4.3.1. Matrix interferences: Internal standards will be used to correct for some matrix interferences.

4.3.2. A representative sample(s) of the project or projects being analyzed will be screened to avoid using an analyte as an internal standard that is present in the samples at a concentration greater than 10% of the internal standard concentration.

4.3.3. Internal standards should be added at a level to give approximately 100,000 - 2,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ±50 amu of the mass of the affected analyte.

4.3.4. Severe matrix effects are monitored by comparing the internal standard intensities of the sample to the internal standard intensities of the initial calibration blank. If any internal standard intensity is less than 30% or greater than 120% of the internal standard intensity of the calibration blank, a fivefold dilution will be performed on the sample to correct for severe matrix effects, and the sample reanalyzed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S

Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.1.2. This instrument produces RF energy. People with pacemakers should not go near the instrument while it is in operation.

5.1.3. Some extractions and digestions use hydrofluoric acid, which means that some samples to be analyzed are in an HF solution. The labels for all such extracts and digestates will be highlighted in yellow. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S staff and the local supplement to the Corporate Safety Manual for guidance. Anyone working with HF must receive special training before starting work. Staff members who work around HF should also receive this training. HF acid solutions may not be used for any purposes except as prescribed in this or other TestAmerica Sacramento SOPs. Processes involving HF acid are classified as high-risk activities. Personnel involved must wear a face shield in addition to safety glasses or goggles.

WARNING: The TestAmerica Sacramento Emergency Response Team must be activated for any suspected exposure to HF liquid or fumes. After local emergency response, the victim will be transported to the UCD Medical Center Emergency Room. Appendix 1 is a detailed first aid plan for working with HF.

5.1.3.1. 2.5% calcium gluconate gel should be applied copiously and gently massaged into burn sites after rinsing under cold running water for one minute to remove HF acid from the surface of the skin. The person massaging should wear appropriate protective gloves. If the victim is conscious, they should consume small quantities of calcium or magnesium containing liquids, such as milk, Maalox, or Mylanta. If the burn site is too large to massage with calcium gluconate gel, cover it with gauze soaked in an Epsom salt solution (1 cup to 1 quart of cold water). Keep the wraps saturated with the Epsom salt solution.

5.1.3.2. Some metals react with HF to produce flammable hydrogen gas.

5.1.3.3. Glass reacts with HF to produce toxic silicon tetraflouride.

5.1.3.4. HF is classified as a poison, and must be stored in a locked cabinet when not in use.

5.1.3.5. Whenever HF is in use in a laboratory, a decontamination solution will be prepared before starting work. Take a small bucket (2-3 gallons) and fill it about 2/3-3/4 full with an Epsom salts solution (1/2 cup of Epsom salts per quart of water). Before removing gloves or handling anything else that HF on the gloves might be transferred to, carefully dip one hand at a time into the Epsom salts solution for a few seconds. Do not put the hand any deeper than necessary to bring the solution level to within 1-2 inches of the glove cuff. The Epsom salts solution will be prepared each day prior to beginning work with HF, and disposed at the end of the day down the laboratory sink.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide satisfactory protection.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

5.2.1. Although no hazardous materials are used during this process, the following is a list of the materials which might be found in the digestates, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

5.2.2. A complete list of materials used in the method can be found in the reagents

and materials section of the extraction/digestion SOP related to the samples in question.

5.2.3. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (1)	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid (1)	Poison Corrosive Dehydrator	3 PPM - TWA	Corrosive to the skin and eyes. Contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. Severely corrosive to the respiratory tract. Permanent eye damage may occur. THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

Preventive maintenance schedules for instrumentation may be found below.

INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS)[1]	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics. Measure quartz torch for proper alignment when removed and cleaned. Clean spray chamber and nebulizer. Clean all filters and fans. Check chiller coolant level. Check and drain oil mist eliminator on roughing pumps.	As Needed
	Check sample waste container level. Check quartz torch condition. Check RF coil. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing. Check condition of sampler and skimmer cones. Check oil level of roughing pumps.	Daily[2]
	Replace oil in roughing pumps.	Every 2-3 Months

6.1. ICP-MS Instrument, either:

6.1.1. Perkin Elmer/Sciex ELAN 6000 ICP-MS or equivalent, utilizing Elan software, version 2.3.2 or equivalent.

6.1.2. Agilent 7700x (G3281A) ICP-MS, equipped with:

6.1.2.1. Autosampler (ASX-5000 Series)

6.1.2.2. Integrated Sample Introduction System (ISIS)

6.1.2.3. MassHunter Software G7201B version B.01.03, Build 393.8

6.2. Argon gas: High-purity grade (99.99%).

6.3. Helium gas: High-purity grade (99.99%) for use with Agilent Instrument.

6.4. Hydrogen gas: High-purity grade (99.99%) for use with Agilent Instrument

6.5. Water re-circulator / chiller.

6.6. Adjustable air displacement pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes, 13 x 100 mm polystyrene or polypropylene.

7. REAGENTS AND STANDARDS

Use reagent grade chemicals in all tests. “Certificates of Analysis” should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate

and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.1. Reagent water is produced by a Millipore Nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.3. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.4. Instrument rinse: Add 2000 mL of reagent water to the 4000 mL rinse container. Add 80mL of concentrated HNO_3, 40mL of concentrated HCl, then dilute to volume with reagent water.

7.5. Stock standards are purchased as custom multi-element mixes and as single-element solutions. These must be plasma grade standards.

7.5.1. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.

7.5.2. Upon making a standard, the process must be documented in the standards log book and the standard given a specific ID according to laboratory procedure.

7.5.3. All intermediate and working standards must be labeled with the name of the preparer, the date prepared, the expiration date, acid concentrations, and the standard specific ID.

7.5.4. Standards containing silver must be protected from light. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer.

7.5.5. If no expiration date is provided, the stock solutions may be used for up to one year from opening and must be replaced sooner if verification from an independent source indicates a problem.

7.6. Calibration Standard/Continuing Calibration Standard: see Table 2 for instructions on making this standard.

7.7. Initial Calibration Verification (ICV) standard: see Table 3 for instructions on making this standard.

7.8. Low Level Check Intermediate Standard: see Table 4 for instructions on making this standard.

7.9. ICSA standard: see Table 5 for instructions on making this standard.

7.10. ICSAB standard: see Table 6 for instructions on making this standard.

7.11. Internal Standard A Intermediate: See Table 7 for instructions on making this standard.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.2. All soils must be refrigerated to $3^{o}C \pm 3^{o}C$.

8.3. The analytical holding time for metals by ICP-MS is 6 months from date of collection.

9. QUALITY CONTROL

9.1. Batch - a quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards.

9.1.1. A batch must be prepared within the same time frame.

9.1.2. A method blank (MB) and a laboratory control sample (LCS) must be prepared as a part of every batch.

9.1.3. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/MSD).

9.1.4. An analytical batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One method blank (MB) must be prepared for every 20 samples.

9.2.1. A method blank consists of reagent water processed through all of the steps, and at the same time as the associated samples.

9.2.2. If a method blank exceeds ± the reporting limit for a given analyte then the samples associated with that batch must be re-prepared.

9.2.2.1. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo.

9.2.3. For samples requiring laboratory filtration and preservation, the method blank

must also be filtered and preserved. See policy WS-PQA-003 of the Quality Control Program for further details.

9.2.4. DOD projects including QSM Versions 4.2 and AFCEE Version 4.0: the results of the method blank must be ½ the reporting limit for a given analyte. The same exceptions and corrective actions apply as stated in section 9.2.2.1

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples.

9.3.1. A LCS for water samples consists of reagent water spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. The LCS for solid samples consists of PTFE boiling chips spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples.

9.3.2. The LCS recoveries are evaluated against in-house generated control limits, which are generated per policy WS-PQA-003 and found in the laboratory's LIM system.

9.3.3. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared for the out of control analyte/s except when a LCS exhibits high recovery.

9.3.3.1. For such a case, those samples with analyte concentrations less than the reporting limit can be reported.

9.3.3.2. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3.4. For samples analyzed under DoD QSM Version 4.2, and AFCEE criteria, there is no exception for reporting "ND" samples for a high LCS.

9.3.5. Random marginal exceedance: For analyte lists of 11-30 elements, 1 LCS random marginal exceedance is allowed. One out of control analyte may be accepted if the result is within 4 standard deviations of the mean. The QA department must validate all marginal exceedance incidents. A NCM must be filed when reporting a marginal exceedance.

9.4. The lower limit of quantitation (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ± 30% of their true value. This check should be

used to both establish and confirm the lowest quantitation limit. The LLQC going through preparation procedure will be called LLCS for TALs identification.

9.5. A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples.

For method 200.8 analysis, an MS is prepared and analyzed for every 10 field samples, i.e., for a batch of 10 samples or fewer, one MS is required, for batches of 11 -20 samples, 2 MS are required. Compare the percent recovery to the method limits of 70-130%.

9.5.1. MS/MSD pairs are aliquots of a selected field sample spiked with all of the analytes of interest at known concentrations The MS/MSD pair must be processed in the same manner and at the same time as the associated samples.

9.5.2. Compare the percent recovery and relative percent recoveries to that in the historically generated control limits. These control limits can be found in the laboratory's LIM system and are generated per policy WS-PQA-003.

9.5.2.1. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS.

9.5.2.2. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

9.5.3. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a MS/MSD pair, and batch precision is required by the client or program.

9.5.3.1. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 of the Quality Control Program for further details.

9.5.4. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for MS/MSD analysis.

9.5.5. If the amount of an analyte found in the un-spiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply.

9.6. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard (ICV) immediately upon completion of instrument calibration.

9.6.1. This standard must be at a concentration different from that used to calibrate the instrument and different from the CCV standard.

9.6.2. The ICV must fall within +/- 10% of the true value of the standard solution. Table 3 shows the concentration of each standard target analyte in the ICV.

9.6.2.1. An ICB (initial calibration blank) prepared the same as the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.

9.6.2.2. The ICB result must fall within +/- the reporting limit from zero.

9.6.3. If either the ICV or ICB fail to meet acceptance criteria the analyst should determine the cause and proceed with corrective action if warranted. This may require re-running the ICV or ICB, repouring and rerunning either the CCV or ICB, or remaking and reanalyzing the ICV or ICB. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the ICV and ICB. The analyst may also recalibrate and rerun the ICV and ICB to verify the calibration.

9.6.4. AFCEE Version 4.0: The ICB and CCB results for all analytes must be less than or equal to 2X the MDL.

9.6.5. QSM Version 4.2: The ICB and CCB results for all analytes must be less than or equal to the LOD (limit of detection).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard, the continuing calibration standard, after every 10 samples and at the end of an analytical run.

9.7.1. Ten samples include any analysis that registers with a result, even if not used.

9.7.2. For method 6020 analysis, the calibration standard is also used as the CCV. The CCV result must fall within +/-10% of the true value for that solution.

9.7.3. A CCB (continuing calibration blank) is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero.

9.7.4. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples.

9.7.5. Results for given analytes may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs for the analytes.

9.7.6. If a mid-run CCV or CCB fails for any number of analytes, the possible cause of failure should be investigated, and all samples analyzed since the last acceptable CCV and or CCB must be reanalyzed for the unacceptable analytes.

9.7.7. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample.

9.7.8. During the course of an analytical run, the instrument may be “resloped” or recalibrated on a CCV/CCB pair to correct for instrument drift. A recalibration must then be followed immediately by a new analysis of a CCV/CCB pair before any further samples may be analyzed. Any samples bracketed (including prior to the reslope) by a failed CCV/CCB must be reanalyzed for the unacceptable analytes.

9.8. Low Level Check Standard (LLSTD, LLICV, or LLCCV): Following analysis of the ICV/ICB pair the low level check standard/s is analyzed spiked with concentrations of each analyte of interest at or below the reporting limit. For method 6020A, it is also recommended to analyze the standard after samples (refer also to Section 10.9 for details and criteria).

9.8.1. The percent recovery of the low level standard results must be +/-50% of the true concentration for all standards analyzed that meet or exceed the reporting limit.

9.8.2. QSM Version 4.2 and AFCEE Version 4.0: Low level check standard recovery must be ± 20%.

9.8.3. For Method 6020A the LLICV results must be ± 30% of the true concentration for all standards analyzed that meet or exceed the reporting limit.

9.8.4. If the recovery is out of control, the analyst should determine the cause and proceed with corrective action. Appropriate corrective actions include rerunning the LLSTD, repouring and rerunning the LLSTD, or remaking and reanalyzing the LLSTD. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the LLSTD. The analyst may also recalibrate and rerun the ICV/ICB and other QC necessary for the run.

9.9. For all analyses run under DOD criteria (QSM, AFCEE), linear range standards (LRSTD) are analyzed after the completion of calibration.

9.9.1. The linear range standards contain the analytes of interest at high levels.

9.9.2. The acceptance criterion for linear range standards is ± 10% of the true value.

9.9.2.1. If this criterion is not met for a given analyte/s, a lower concentration standard should be analyzed such that the criterion is met.

9.9.3. Once linear range has been established through the analysis of the linear range standards, no value can be reported that exceeds the concentration of the standards.

9.9.3.1. If sample concentrations exceed that of the linear range standards for any given analyte, the sample requires dilution at a level where all analytes fall below the level of the linear range standards.

9.10. Interference Check Analysis (ICSA/ICSAB): Interference check standards ICSA and ICSAB are analyzed directly after analysis of the low level check standard.

9.10.1. The ICSA/ICSAB are essentially remnants of method 6010 where the analytes spiked in the standard at high levels are considered interferents. For method 6020 these are not normally interferents, but represent analytes that might be seen at higher levels in real world samples.

9.10.2. ICSA (interference check standard A) contains only the high level analytes (interferents) without any spiking of the analytes more notably determined at lower relative concentrations. See Table 5 for the composition of the ICSA.

9.10.3. ICSA is also spiked with 10 ppm bromide to evaluate the correction required for bromide interference on selenium 82. Once the ICSA has been analyzed, the correction factor should be adjusted such that the result for selenium 82 is less than +/- the reporting limit.

9.10.4. Method 6020 does not place any acceptance criteria on ICSA, but only requires that it be analyzed.

9.10.4.1. For standard work, the ICSA concentration of non-spiked analytes should be less than two times the reporting limit to continue analysis. If it exceeds this limit, an NCM should be filed to explain the reason. Acceptable narrations include:

- All associated samples have high levels of the analyte (greater than 10x the level found in the ICSA)
- All associated samples are non-detect for the analyte.

Additional corrective actions include rerunning, repouring, and remaking and rerunning the ICSA.

9.10.4.2. AFCEE Version 4.0 requires the concentration of non-spiked analytes be less than 2X the MDL to continue analysis. Refer to Section 9.10.4.1 (above) for corrective action.

9.10.4.3. QSM 4.2 requires that the absolute value of the concentration of non-spiked analytes be less than the LOD to continue analysis. Refer to Section 9.10.4.1 (above) for corrective action.

9.10.5. The acceptance criteria for all of the methods does not apply when trace impurities are validated by the Certificate of Analysis for the ICSA solution that cause the non-spiked analytes to show positive concentrations outside of acceptance criteria. In such a case, a NCM is required that identifies the impurities and their certified concentrations in the standard.

9.10.6. ICSAB (interference check sample AB) is the same as ICSA, but all of the non-interfering analytes are spiked into the ICSA at the concentrations shown in Table 6.

9.10.7. The ICSAB results for the low level (non-interfering) analytes must fall within 80 – 120% (79.5 – 100.49 with rounding) of the true value. If the ICSAB does not meet these criteria for any analyte, the analyst should determine the cause and proceed with corrective action. Acceptable corrective actions include rerunning the ICSAB, repouring and rerunning the ICSAB, or remaking and reanalyzing the ICSAB. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the ICSAB. The analyst may also recalibrate and rerun the ICV/ICB and other QC necessary for the run.

9.10.8. For Method 6020A verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the ICSA and ICSAB.

9.11. Dilution test (Serial Dilution) – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.

9.11.1. One sample per preparation batch must be processed as a dilution test.

9.11.2. The test is performed by running a sample at a 5X (1+4) dilution.

9.11.3. Samples identified as field blanks cannot be used for dilution tests.

9.11.4. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 100X the MDL.

9.11.4.1. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.12. Post-digestion spikes: One sample in every batch may be spiked with all of the elements of interest at the instrument following sample preparation. The matrix spiking solution is generally used as the post-digestion spike resulting in the same concentration spike level as the matrix spikes.

9.13. Internal standards are added continuously to all analyses in an analytical run.

9.13.1. For method 6020, the internal standard (IS) intensities in samples must be within 30 to 120% of the IS intensities for the initial calibration blank, although suppression influencing sample results could be expected at levels less than 60-70% recovery. If the 30 – 120% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

9.13.2. For method 6020A, the internal standard (IS) intensities in samples must be greater than 70% of the IS intensities for the initial calibration standard. If the intensity in the sample is less than 70% of the intensity in the initial calibration standard, ensure that the instrument has not drifted by checking the intensities in the nearest calibration blank. If low intensities are also present in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, and reanalyze the affected samples. If drift has not occurred, remove matrix effects by diluting the sample. Dilute at least 5x and reanalyze. Repeat if the first dilution is not sufficient to ameliorate the matrix effects. Narrate any findings.

9.13.3. For Method 200.8 the internal standard (IS) intensities in the samples must be within 60-125 % of the IS intensities for the initial calibration blank. If the 60 – 125% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

9.13.4. The internal standard intensities of the ICV/CCVs and ICB/CCBs should be within 80 – 120% recovery relative to the internal standard intensities of the calibration blank.

9.13.4.1. If this criterion is not met, the analyst must recalibrate (or reslope) the instrument and rerun all samples associated with the out of control QC. Only analytes associated with the out of control internal standard require re-analysis.

9.13.5. Table 7 shows the composition of the internal standard A solution. This standard is diluted further at 2X or 3X prior to analysis.

9.13.6. The internal standard raw intensity counts must be printed on the raw data. The percent recovery of each internal standard relative to the calibration blank or the blank associated with a previous re-slope is printed on the raw data.

9.14. High Level Continuing Calibration Verification (High Level CCV) – This standard is used to verify the instrument's dual detector calibration.

9.14.1. The High Level CCV is analyzed at the beginning of the run to demonstrate whether the instrument is accurately measuring concentration at concentrations near the upper limit of the linear range. If the values are greater than ± 20%D a new dual detector calibration may be required.

9.14.2. Should values in the High Level CCV exceed the ± 20%D criteria, only values less than or equal to the nominal values of the mid-level continuing calibration may be reported.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Instrument start-up

10.2.1. Follow the instrument start-up procedure outlined in the Perkin Elmer Elan 6000 Operator's Manual and Agilent 7700X Operator's Manual.

10.3. Instrument Tuning

10.3.1. Perkin Elmer

10.3.1.1. Aspirate a 10 ppb tuning solution containing Li, Co, In, and Tl.

10.3.1.2. A mass calibration and resolution checks must be documented and included as part of the raw data package. If the following conditions are not met, re-tune the instrument.

10.3.1.3. Resolution must be < 0.9 amu full width at 10% peak height. The Elan 6000 resolution is maintained at approximately 0.7 amu.

10.3.1.4. Mass calibration must be within ± 0.1 amu from the true value.

10.3.1.5. A daily performance check must be performed as part of the daily "tune". This uses the same solution as in section 10.3.1, and this also contains Mg, Rh, and Ba. The elements Li, Co, In, and Tl must be below 5% RSD. Rh must be at or above 200,000 counts. The CeO and Ba^{++} intensity readouts must be below 0.035 which shows the oxides and doubly charged ions are below 3.5%. .If any of these conditions are not met, maintenance or optimization should be performed until these specifications are met, or an NCM written to explain the affect on the analysis and clients results.

10.3.2. Agilent 7700X

10.3.2.1. A US EPA Tune Check Sample must be performed in the Helium [He] mode and Hydrogen [H2] mode. The US EPA Tune Check Sample contains Ba, Be, Ce, Co, In, Li, Mg, Pb, Rh, Tl, U and Y at 10 ppb. In the [He] mode the RSD% for mass 24, 59, 115, and 208 must be below 5%. In the [H2] mode mass 9, 24, 59, 115, and 208 must be below 5%.

10.3.2.2. Resolution must be < 0.9 amu full width at 10% peak height. The Agilent 7700X resolution is maintained at approximately 0.75 amu.

10.3.2.3. Mass calibration must be within ± 0.1 amu from the true value.

10.4. Initial calibration must be performed daily and each time the instrument is set up.

10.4.1. Calibration consists of a blank and a single calibration standard. Table 2 shows the concentration of all analytes in the calibration standard. Use the average of three integrations for both calibration and sample analyses. Initial calibration acceptance is based upon the ICV falling within +/- 10% recovery from the true value.

10.5. Following calibration, an ICV/ICB pair is analyzed to confirm the accuracy of the calibration (section 9.5).

10.6. Low level check standards are analyzed to confirm the sensitivity of the analytical run at the reporting limit for the samples (section 9.7).

10.7. For DOD, linear range standards are analyzed after the completion of the calibration as per section 9.8.

10.8. Continuing calibration verifications, CCV/CCB pair, are analyzed every ten (10) samples and at the end of the analytical sequence as per section 9.6.

10.9. For Method 6020A, the low level continuing calibration verification (LLCCV) standard should also be analyzed at the end of each analysis batch. A more frequent LLCCV analysis, i.e., every 10 samples may be necessary if low-level sample concentrations are anticipated and the system stability at low end of the calibration is questionable. The acceptance criteria for the LLCCV standard should be ± 30% of its true value.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All samples require a preparation prior to analysis depending on the matrix being evaluated. Preparation of each batch requires a method blank, LCS, and matrix spikes and/or matrix spike duplicates. The concentration of analytes in the LCSs and matrix spikes are listed in the individual preparation methods.

11.2.1. For method 6020 and 6020A soils analysis, EPA method 3050B is used to prepare all samples.

11.2.2. For method 200.8, 6020, 6020A aqueous samples, and TCLP leachate samples are prepared using the total recoverable digestion procedure, method 3005A (WS-IP-0001).

11.2.3. For method 200.8, 6020, 6020A dissolved samples, and STLC are prepared using the matrix matching procedure (SOP WS-IP-0008).

11.3. Before setting up an analytical run, read the QAS for all of the lots being analyzed. The QAS is created by the project managers and defines any special criteria required for client specific analyses.

11.4. An instrument backlog is generated from the TALs data system. This backlog lists the Job numbers that were assigned to each batch of samples. The backlog also shows project due date, number of samples and sample matrix. The instrument backlog aids the analyst in deciding the lot IDs to analyze on a given day.

11.5. An analytical run will consist of all customer samples and quality control samples analyzed under a daily initial calibration. Each new initial calibration will begin a new analytical run.

11.6. Using utility software, the analyst can generate a sample run log directly from the instrument software.

11.7. In order to use the ICP-MS data upload program into TALs, the following naming conventions must be followed:

11.7.1. Samples are identified by the 5 character work order number.

11.7.2. Quality control parameters have a suffix after their sample ID...

Parameter	Suffix
Method Blanks	MB
LCS	LCS
LCS Duplicate	LCSD
Matrix Spike	MS
Matrix Spike Duplicate	MSD
Sample Duplicate	DU
Serial Dilution	SD
Post-digestion Spike	PDS

11.8. Set-up

11.8.1. Open a new dataset using the date and instrument in the title. For instance the first run (A) on instrument 2 on JAN 1, 2013 would be 070113A2.

11.8.2. Open the appropriate method if one already exists or create a new one for the analytes to be quantitated in the run. A method with all of the analytes has been generated and can be modified to the specific analytes required throughout any given analytical run.

11.8.3. All masses which could affect data quality should be monitored to determine potential interferences. Secondary masses are evaluated per SW 846 protocols.

11.8.4. Internal standards are added to all standards and samples by the instrument peristaltic pump and a mixing tube prior to analysis.

11.8.5. Use of an existing autosampler table is suggested. A read delay of 45 to 60 seconds is used between all analyses.

11.9. The order of analysis for the initial QC samples and calibration should be:

1. Rinse
2. CAL Blank
3. STD1 (Calibration Standard)
4. ICV (Second source, must be ± 10% of true value)
5. ICB
6. LLSTD1
7. LLSTD2, etc
8. ICSA (Interference check solution with Bromine, for Selenium. ± RL for Se.)
9. ICSAB (Interference check solution, ± 20% of true value)
10. LRSTD1
11. LRSTD2, etc
12. Rinse (up to 3 rinses)
13. CCV1
14. CCB1
15. CCV2
16. CCB2

11.10. To continue the analytical run, add an additional 10 runs followed by CCV/CCB, and repeat for up to 24 hours.

11.11. Samples must be diluted and reanalyzed for any elements which are present at concentrations exceeding 90% of the upper linear range limit as determined in quarterly linear range studies.

12. CALCULATIONS/DATA REDUCTION

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{Found(ICV)}{True(ICV)} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{Found(CCV)}{True(CCV)} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \times \left(\frac{SSR - SR}{SA} \right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100 \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg / L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight in Kg of wet sample digested

S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \times \left(\frac{Found(LCS)}{True(LCS)} \right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%\ \text{Difference} = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)

S =Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported in accordance with the TestAmerica Sacramento significant figure policy (WS-PQA-004).

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits (Method 6020) or the static recovery limits of 85-115% (Method 200.8).

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acid waste consisting of unused sample, analyzed sample solution and rinse solution. This is collected in four-gallon plastic carboys. When full or after no more than one year, the waste is dumped into an acid waste drum in the H-3 closet. When full to no less than one inch and no more than four inches of the top, or after no more than 75 days, the drum will be transferred to the waste collection area for disposal.

15.2. Miscellaneous disposable glassware, plastic vials with snap top caps, autosampler tubes and similar solid waste. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, September, 1994.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV, Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, February 2007.

16.3. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111), Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma – Mass Spectrometry, Revision 5.4, 1994

16.4. EPA Method 200.8 EMSL office of Research & Development, Cincinnati, OH (Draft Method, Revision 4.3, August 1990).

16.5. Perkin Elmer/Sciex ELAN model 6000 Users Manual

16.6. Agilent Series 7700 ICP-MS Users Manual

16.7. Department of Defense Quality Systems Manual for Environmental Laboratories (QSM), Version 4.2, October 2010.

16.8. AFCEE QAPP, Version 4.0, February 2005

16.9. Recommended ICP-MS References

16.9.1. K. E. Jarvis, A. L. Gray, and R. S. Honk, Handbook of Inductively Coupled Plasma Mass Spectrometry; Chapman & Hall, New York 1992.

16.9.2. A. R. Date and A. L. Gray, Applications of Inductively Coupled Mass Spectrometry, Chapman & Hall, New York 1989. ISBN # 0-41201721-0

17. METHOD MODIFICATONS

Deviations from Source Method and Rationale

17.1. As a conservation of space, the raw data will only contain the mean and standard deviation results on standard ELAN summary report. As long as the standard deviation data is shown, the analyst can make decisions on replicate quality on the standard ELAN summary report.

17.2. This SOP may be used to analyze for elements not included in Method 6020, as long as appropriate QC samples spiked with the non-routine analytes are run with acceptable results. Clients should be notified that we do not carry method performance indicators for these analytes.

17.3. For reporting consistency, an ICB/CCB is acceptable if the result is <RL. Method 6020 states that the results of the calibration blank (CCB) are to be less than 3x the IDL. If not, terminate the analytical sequence, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica Sacramento has adopted an absolute control limit of +/- RL from zero for calibration blank criteria for standard operations. Exceptions are defined in this SOP.

17.4. The ICSA and ICSAB are being made as needed with an expiration date of 5 months or less depending on stock solutions expiration dates. Method 6020 states that the ICSA and ICSAB need to be prepared fresh weekly from ultra pure reagents. TestAmerica Sacramento has adopted to make these standards as needed due to the stability of the purchased stock solutions (manufacturer's expiration date or <1 year).

18. ATTACHMENTS

Tables referenced in the body of the SOP

18.1. Table 1 -Standard Target Analyte List

18.2. Table 2 -Calibration and Calibration Check Standard Preparation

18.3. Table 3 -Initial Calibration Verification Standard

18.4. Table 4 -Low Level Check Intermediate Standard Preparation

18.5. Table 5 -Interference Check Solution A

18.6. Table 6 -Composition of the ICSAB Standard

18.7. Table 7 -Internal Standard A Stock

18.8. Table 8 -Common Molecular Ion Interferences in ICPMS

18.9. Table 9 -Recommended Analytical Isotopes and Additional Masses (Elements of Interest)

18.10. Table 10 -Recommended Analytical Isotopes and Additional Masses (Rare Earth Elements and other Elements)

18.11. Table 11 - Elemental Equations used in Calculations

19. REVISION HISTORY

19.1. WS-MT-0001, Revision 3.8, Effective 12/12/2014

19.1.1. Added EPA Method 200.8 to Scope and Application Sections 1,1 and 1,3.

19.1.2. Section 6.4 – Changed Argon gas to Hydrogen gas.

19.1.3. Added Section 9.13.3 – "For Method 200.8 the internal standard (IS) intensities in the samples must be within 60-125 % of the IS intensities for the initial calibration blank. If the 60 – 125% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the

upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

19.1.4. Added Section 10.3.2 for Agilent 7700X instrument parameters.

19.1.5. Added the following paragraph following Section 9.5 -"For method 200.8 analysis, an MS is prepared and analyzed for every 10 field samples, i.e., for a batch of 10 samples or fewer, one MS is required, for batches of 11 -20 samples, 2 MS are required. Compare the percent recovery to the method limits of 70-130%.

19.1.6. Added the following to the end of Section 13.3.2 – "or the static recovery limits of 85-115% (Method 200.8)"

19.1.7. Editorial changes..

19.2. WS-MT-0001, Revision 3.7 Effective 08/29/2014

19.2.1. Added Section 9.10.8, "For Method 6020A verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the ICSA and ICSAB.)

19.2.2. Editorial changes.

19.3. WS-MT-0001, Revision 3.6, Effective 09/30/2013

19.3.1. Revised Section 9.5.3, 9.7.3 (number changed from 9.7.2) and 9.9.4 to reflect how analytical runs and decision making proceeds.

19.3.2. Removed Section 10.4 (Screening for Internal Standards)

19.3.3. Added Section 11.11 (regarding dilutions for analytes > linear range study)

19.3.4. Added Section 9.14 (High Level CCV) to reflect current practice to improve data quality.

19.3.5. Added references to method 6020A in Sections 1.3, 11.2, and 16.2

19.3.6. Added reference to preventive maintenance schedules in Section 6, and added Table 12 (Preventative Maintenance).

19.3.7. Deleted references to AFCEE 3.1 in Sections 9.3

19.3.8. Inserted Section 9.4 (low level QC Check sample)

19.3.9. Inserted Section 9.8.3 regarding the low-level standard frequency and criteria for method 6020A.

19.3.10. Sections 9.6.3, 9.8.4, 9.10.4.1, and 9.10.7 Clarified the corrective action procedures.

19.3.11. Section 10.3, removed elements not required in the 6020/6020A tuning solution.

19.3.12. Added Section 10.8, low-level calibration standard at the end of the run.

19.3.13. Section 11.4 through 11.7, minor edits to reflect procedure using TALS vs. the older LIMs.

19.3.14. Editorial changes.

19.4. WS-MT-0001, Revision 3.5, Effective 07/05/2013

19.4.1. Modified Section 10.3.3 to follow what is in the method regarding tuning.

19.4.2. Editorial changes.

19.5. WS-MT-0001, Revision 3.4, Effective 09/14/2012

19.5.1. Removed requirement for making ICSA and ICSAB standards weekly from Section(s) 7.9 and 7.10.

19.5.2. Removed references to AFCEE 3.1.

19.5.3. Editorial changes.

19.6. WS-MT-0001, Revision 3.3, Effective 06/10/2011

19.6.1. Inserted Cd (mass 114) in Table 1.

19.6.2. Deleted reference to 6020A in Method heading and reference (Section 19.2).

19.6.3. Removed Section 6.5: Class A volumetric pipettes.

19.6.4. Section 9.11: replaced “must be spiked” with “may be spiked”.

19.6.5. Section 10.4.1: replaced “representative samples must be screened” with “it is recommended that soil samples be screened”.

TABLE 1			
Method 6020 Standard Target Analyte List			
Element	**Symbol**	**Aqueous RL mg/L**	**Soil RL mg/Kg**
Aluminum	Al	0.05	5.0
Antimony	Sb	0.002	0.2
Arsenic	As	0.002	0.2
Barium	Ba	0.001	0.1
Beryllium	Be	0.001	0.1
Boron	B	0.05	5.0
Cadmium	Cd (mass 111)	0.001	0.1
Cadmium	Cc (mass 114)	0.001	0.1
Calcium	Ca	0.05	30
Chromium	Cr	0.002	0.2
Cobalt	Co	0.001	0.1
Copper	Cu	0.002	0.2
Iron	Fe	0.05	5.0
Lead	Pb	0.001	0.1
Lithium	Li	0.01	1.0
Magnesium	Mg	0.05	5.0
Manganese	Mn	0.001	0.2
Molybdenum	Mo	0.002	0.2
Nickel	Ni	0.002	0.2
Phosphorous	P	0.05	20
Potassium	K	0.05	5.0
Selenium	Se (mass 82)	0.002	0.2
Selenium	Se (mass 78)	0.003	0.3
Silver	Ag	0.001	0.1
Sodium	Na	0.05	50
Strontium	Sr	0.005	0.5
Thallium	Tl	0.001	0.1
Tin	Sn	0.01	2.0
Titanium	Ti	0.005	0.5
Vanadium	V	0.01	1.0
Zinc	Zn	0.005	1.0

Table 2				
Calibration Standard and Continuing Calibration Verification Standard				**Final Volume = 500 mL**
Element	**Stock Standard ID**	**Stock Standard Concentration (mg/L)**	**Spike Volume (mL)**	**Final Concentration mg/L**
Aluminum	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Antimony	CPI-ICPMS-1B	5.0	5.0	0.050
Arsenic	CPI-ICPMS-1B	10	5.0	0.10
Barium	CPI-ICPMS-1B	10	5.0	0.10
Beryllium	CPI-ICPMS-1B	10	5.0	0.10
Boron	CPI-ICPMS-1B	50	5.0	0.10
Cadmium	CPI-ICPMS-1B	10	5.0	0.10
Calcium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Chromium	CPI-ICPMS-1B	10	5.0	0.10
Cobalt	CPI-ICPMS-1B	10	5.0	0.10
Copper	CPI-ICPMS-1B	10	5.0	0.10
Iron	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Lead	CPI-ICPMS-1B	10	5.0	0.10
Lithium	CPI-ICPMS-1B	10	5.0	0.10
Magnesium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Manganese	CPI-ICPMS-1B	10	5.0	0.10
Molybdenum	CPI-ICPMS-1B Spex ZCAL-34	10 20	5.0 2.5	0.20
Nickel	CPI-ICPMS-1B	10	5.0	0.10
Phosphorous	Spex ZCAL-34	1000	2.5	5.0
Potassium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Selenium	CPI-ICPMS-1B	10	5.0	0.10
Silver	CPI-ICPMS-1B	5.0	5.0	0.050
Sodium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Strontium	CPI-ICPMS-1B	10	5.0	0.10
Thallium	CPI-ICPMS-1B	5.0	5.0	0.050
Tin	CPI-ICPMS-1B	10	5.0	0.10
Titanium	CPI-ICPMS-1B Spex ZCAL-34	10 20	5.0 2.5	0.20
Vanadium	CPI-ICPMS-1B	10	5.0	0.10
Zinc	CPI-ICPMS-1B	10	5.0	0.10
Sulfur	Spex ZCAL-34	1000	2.5	5.0
Carbon	Spex ZCAL-34	2000	2.5	10
Chloride	Spex ZCAL-34	7200	2.5	36

TABLE 3			
Method 6020 Initial Calibration Verification Standard Stock: CPI CALLAB-3 Spike Volume: 4.0 mL Final Volume: 500 mL			
Element	**Concentration mg/L**	**Element**	**Concentration mg/L**
Aluminum	0.8	Manganese	0.08
Antimony	0.04	Molybdenum	0.08
Arsenic	0.08	Nickel	0.08
Barium	0.08	Phosphorous	0.8
Beryllium	0.08	Potassium	0.8
Boron	0.8	Selenium	0.08
Cadmium	0.08	Silver	0.04
Calcium	0.8	Sodium	0.8
Chromium	0.08	Strontium	0.08
Cobalt	0.08	Thallium	0.04
Copper	0.08	Tin	0.08
Iron	0.8	Titanium	0.08
Lead	0.08	Vanadium	0.08
Lithium	0.08	Zinc	0.08
Magnesium	0.8		

Table 4				
Low Level Check Intermediate Standard - Method 6020			**Final Volume = 500 mL**	
Element	**Stock Standard ID**	**Stock Standard Conc'n (mg/L)**	**Spike Volume (mL)**	**Final Conc'n (mg/L)**
Aluminum	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Antimony	CALLAB-3	5.0	0.5	0.005
Arsenic	CALLAB-3	10	0.5	0.01
Barium	CALLAB-3	10	0.5	0.01
Beryllium	CALLAB-3	10	0.5	0.01
Boron	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Cadmium	CALLAB-3	10	0.5	0.01
Calcium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Chromium	CALLAB-3	10	0.5	0.01
Cobalt	CALLAB-3	10	0.5	0.01
Copper	CALLAB-3	10	0.5	0.01
Iron	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Lead	CALLAB-3	10	0.5	0.01
Lithium	CALLAB-3 Single	100 1000	0.5 0.02	0.05
Magnesium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Manganese	CALLAB-3	10	0.5	0.01
Molybdenum	CALLAB-3	10	0.5	0.01
Nickel	CALLAB-3	10	0.5	0.01
Phosphorous	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Potassium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Selenium	CALLAB-3	10	0.5	0.01
Silver	CALLAB-3	5.0	0.5	0.005
Sodium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Strontium	CALLAB-3	10	0.5	0.01
Thallium	CALLAB-3	5.0	0.5	0.005
Tin	CALLAB-3 Single	100 1000	0.5 0.045	0.1
Titanium	CALLAB-3 Single	100 1000	0.5 0.02	0.05
Vanadium	CALLAB-3 Single	100 1000	0.5 0.045	0.1
Zinc	CALLAB-3 Single	100 1000	0.5 0.02	0.05

TABLE 5			
*** Method 6020 Interference Check Solution A** **Stock: Spex ZCAL-34 Spike volume: 10 mL** **Bromide 1000 mg/L stock Spike volume: 1.0 mL** **Final Volume: 100 mL**			
Element	**Concentration mg/L**	**Element**	**Concentration mg/L**
Br^-	100	P	100
Al	100	S	100
Ca	100	C	200
Fe	100	Cl^-	720
K	100	Mo	2.0
Mg	100	Ti	2.0
Na	100		

*** Make new standard weekly**

TABLE 6 Composition of the ICSAB Standard			
Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.05	Na	100.1
Al	100.1	Ni	0.10
As	0.10	Pb	0.10
B	0.10	Sb	0.05
Ba	0.10	Se	0.10
Be	0.10	Si	0.10
Ca	100.1	Sn	0.10
Cd	0.10	Sr	0.10
Co	0.10	Ti	2.0
Cr	0.10	Tl	0.05
Cu	0.10	U	0.10
Fe	100.1	V	0.10
K	100.1	Zn	0.10
Li	0.1	P	100
Mg	100.0	S	100
Mn	0.10	C	200
Mo	2.10	Cl-	720

Table 7			
Internal Standard A Stock - Method 6020		**Final Volume = 4000 mL**	
Element	**Stock Standard Concentration (mg/L)**	**Spike Volume (mL)**	**Final Concentration (mg/L)**
Lithium	500	80	10
Scandium	1000	8.0	2.0
Germanium	10000	3.0	7.5
Indium	1000	3.0	0.75
Thullium	1000	2.0	0.5

TABLE 8[1]					
COMMON MOLECULAR ION INTERFERENCES IN ICP-MS					
Molecular Ion	**Mass**	**Element Interferences[2]**	**Molecular Ion**	**Mass**	**Element Interferences[2]**
BACKGROUND MOLECULAR IONS					
NH^+	15		$^{38}ArH^+$	39	
OH^+	17		$^{40}ArH^+$	41	
OH_2^+	18		CO_2^+	44	
C_2^+	24		CO_2H^+	45	Sc
CN^+	26		ArC^+, ArO^+	52	Cr
CO^+	28		ArN^+	54	Cr
N_2^+	28		$ArNH^+$	55	Mn
N_2H^+	29		ArO^+	56	
NO^+	30		$ArOH^+$	57	
NOH^+	31		$^{40}Ar^{36}Ar^+$	76	Se
O_2^+	32		$^{40}Ar^{38}Ar^+$	78	Se
O_2H_+	33		$^{40}Ar_2^+$	80	Se
$^{36}ArH^+$	37				
MATRIX MOLECULAR IONS – Chloride					
$^{35}Cl0^+$	51	V	$^{37}Cl0H^+$	54	Cr
$^{35}Cl0H^+$	52	Cr	$^{35}Cl0^+$	51	V
$^{37}Cl0^+$	53	Cr	$^{35}Cl0H^+$	52	Cr
$Ar^{35}Cl^+$	75	As	$Ar^{37}Cl^+$	77	Se
MATRIX MOLECULAR IONS – Sulfate					
$^{32}SO^+$	48		$^{34}SOH^+$	51	V
$^{32}SOH^+$	49		SO_2^+, S_2^+	64	Zn
$^{34}SO^+$	50	V, Cr			
$Ar^{32}S^+$	72		$Ar^{34}S^+$	74	
MATRIX MOLECULAR IONS – Phosphate					
PO^+	47		PO_2^+	63	Cu
POH^+	48				
ArP^+	71				
MATRIX MOLECULAR IONS – Group I, II Metals					
$ArNa^+$	63	Cu	$ArCa^+$	80	
ArK^+	79				
MATRIX OXIDES[3]					
TiO	62-66	Ni, Cu, Zn	MoO	108-116	Cd
ZrO	106-112	Ag, Cd			

[1] *Table reference from Method 200.8, Section 13.2.6*

[2] *Method elements or internal standards affected by the molecular ions.*

[3] *Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.*

TABLE 9 RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED [1]			
Isotope	Element of Interest	Isotope	Element of Interest
27	Aluminum [2]	80,**78**,**82**,**76**,**77**,74	Selenium
121,123	Antimony [2]	**107**,**109**	Silver [2]
75	Arsenic [2]	**23**	Sodium [2]
138,137,136,**135**,134,132,130	Barium [2]	203, **205**	Thallium [2]
9	Beryllium [2]	**51**,**50**	Vanadium [2]
114,112,**111**,110,113,116,106, **108**	Cadmium [2]	**66**, **67**, **68**	Zinc [2]
42,**43**,**44**,46,48	Calcium [2]	83	Krypton
52,**53**,**50**,54	Chromium [2]	72	Germanium
59	Cobalt [2]	139	Lanthanum
63,**65**	Copper [2]	140	Cerium
56,**54**,**57**,58	Iron [2]	129	Xenon
206,**207**,**208**	Lead [2]	**118**	Tin
24,**25**,**26**	Magnesium [2]	105	Palladium
55	Manganese [2]	47,**49**	Titanium
202,**200**,199,201	Mercury	125	Tellurium
98,96,92,**97**,94	Molybdenum	69	Gallium
58,**60**,62,**61**,64	Nickel [2]	35,37	Chlorine
39	Potassium [2]		

[1] *From Method 6020 CLP-M, Table 9*

[2] *Element approved for ICP-MS determination by SW846 Method 6020 CLP-M*

NOTE: Isotopes recommended for analytical determination are **bold**.

TABLE 10			
RECOMMENDED ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED			
Rare Earth Elements	**ICPMS Preferred Mass**	**Elemental Equations**	**Additional Masses**
Lanthanum	138.906		
Cerium	139.905		
Praseodymium	140.907		
Neodymium	141.908	-0.125266 * ^{140}Ce	142.910, 144.912
Samarium	151.920	-0.012780 * ^{157}Gd	144.912
Europium	152.929		
Gadolinium	157.924	-0.004016 * ^{163}Dy	156.934
Terbium	158.925		
Dysprosium	163.929	-0.047917 * ^{166}Er	
Holmium	164.930		
Erbium	165.930		
Thulium	168.934		
Ytterbium	173.939	-0.005935 * ^{178}Hf	171.937
Lutetium	174.941		
Other Elements			
Boron	11.009		
Calcium	43.956		
Cesium	132.905		
Gallium	68.926		
Germanium	71.922		
Gold	196.967		
Hafnium	177.944		176.944
Holmium	164.930		
Iridium	192.963		
Lithium	7.016		
Tungsten	183.951	-0001242* ^{189}Os	
Uranium	238.050		
Yttrium	88.905		
Zirconium	238.050		
Niobium	92.906		
Palladium	104.905		
Phosphorus	30.994		
Platinum	194.965		
Rhenium	186.965	-0.099379 * ^{189}Os	
Rhodium	102.905		
Rubidium	84.912		
Ruthenium	101.904	-0.045678 * ^{105}Pd	
Scandium	44.956		
Strontium	87.906		
Tantalum	180.948		
Tellurium	127.905	-0.072348 * ^{129}Xe	
Thorium	232.03		

TABLE 11
ELEMENTAL EQUATIONS USED TO CALCULATE RESULTS

Element	Elemental Equation	Note
Al	(1.000) (^{27}C)	
Sb	(1.000) (^{121}C)	
As	(1.000) (^{75}C) - (3.1278)[^{77}C) - (1.0177)(^{78}C)]	Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.
Ba	(1.000) (^{137}C)	
Be	(1.000) (^{9}C)	
Cd	(1.000) (^{111}C) - (1.073) [(^{108}C) - (0.712) (^{106}C)]	Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present.
Cr	(1.000) (^{52}C)	In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.
Co	(1.000) (^{59}C)	
Cu	(1.000) (^{63}C)	
Pb	(1.000) (^{206}C) + (1.000) (^{207}C) + (1.000) (^{208}C)	Allowance for isotopic variability of lead isotopes.
Mn	(1.000) (^{55}C)	
Mo	(1.000) (^{98}C) - (0.146) (^{99}C)	Isobaric elemental correction for ruthenium.
Ni	(1.000) (^{60}C)	
Se	(1.000) (^{82}C)	Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.
Ag	(1.000) (^{107}C)	
Tl	(1.000) (^{205}C)	
Th	(1.000) (^{232}C)	
U	(1.000) (^{238}C)	
V	(1.000) (^{51}C) - (3.127) [(^{53}C) - (0.113) (^{52}C)]	Correction of chloride inference with adjustment for Cr53. Cl0 51/53 ratio may be determined from the reagent blank.
Zn	(1.000) (^{66}C)	

* Method elements or internal standards affected by the molecular ions.

Title: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A and MCAWW 245.1 [Method SW846 7470A and MCAWW 245.1]

Approvals (Signature/Date):

Thep Phomsopha Inorganics Supervisor	7/02/14 Date	Joe Schairer Health & Safety Manager / Coordinator	7/2/14 Date
Lisa Stafford Interim Quality Assurance Manager	7/7/2014 Date	Robert Hrabak Laboratory Manager	7/7/14 Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1. Method 7470A is applicable to the preparation and analysis of mercury in ground water, wastewater, wastes, wipes, TCLP, SPLP, STLC leachates, and samples from stationary source sampling trains . Method 245.1 is applicable to the determination of mercury in drinking, surface and saline water, domestic and industrial wastes. All matrices require sample preparation prior to analysis. This also applies to digestates prepared by SOPs WS-IP-0007.

1.2. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Samples are digested using a combination of acids and strong oxidizers, converting all forms of mercury to mercuric ions. The samples are treated with hydroxylamine hydrochloride solution to remove excess oxidizing reagents. The digestates are analyzed using Leeman Labs automated mercury analyzers. The mercuric ions are reduced to atomic mercury vapor with the addition of stannous chloride into a gas-liquid separator. The mercury vapor is purged into an absorption cell with nitrogen gas. The relative absorbance of the mercury vapor is detected at a wavelength of 253.7- nm via a lamp/detector system.

2.2. Reporting limit: 0.2 µg/L.

2.3. Calibration Range: 0.2 µg/L to 10 µg/L.

2.4. This analytical method is restricted to use by, or under the supervision of an analyst experience in the operation of cold vapor analysis and the evaluation of the resulting data.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Potassium permanganate, which is used to breakdown organic mercury compounds also eliminates possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. High levels of free chlorine can cause a positive interference. Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 5.0 mL) since chloride is converted to free chlorine during oxidation. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The front half air train samples extracted are in a 2% HF solution. The labels fro all such extracts and digestates should be highlighted in yellow. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S staff and the local supplement to the Corporate Safety Manual for guidance. Anyone working with HF must receive special training before starting work. Staff members who work around HF should also receive this training. HF and solutions may not be used for any purposes except as prescribed in this or other Sacramento SOPs. Processes involving HF acid are classified as high-risk activities. Personnel involved must wear a face shield in addition to safety glasses or goggles.

<u>WARNING</u> The Sacramento Emergency Response Team must be activated for any suspected exposure to HF liquid or fumes. After local emergency response, the victim will be transported to the UCD Medical Center Emergency Room.

5.1.1.1. 2.5% calcium gluconate gel should be applied copiously and gently massaged into burn sites after rinsing under cold running water for one minute to remove HF acid from the surface of the skin. The person massaging should wear appropriate protective gloves. If the victim is conscious, they should consume small quantities of calcium or magnesium containing liquids, such as milk, Maalox, or Milanta. If the burn site is too large for massage with calcium gluconate gel, cover it with gauze soaked in an Epsom salt solution (1 cup to 1 quart of cold water). Keep the wraps saturated with Epsom salt solution.

5.1.1.2. Some metals react with HF to produce flammable hydrogen gas.

5.1.1.3. Glass reacts with HF to produce toxic silicon tetrafluoride.

5.1.1.4. HF is classified as a poison, and must be stored in a locked cabinet when not in use.

5.1.1.5. Whenever HF is in use in a laboratory, a decontamination solution will be prepared before starting work. Take a small bucket (2-3 gallons) and fill it about 2/3 – 3/4 full with an Epsom salts solution. (1/2 cup of Epsom salts per quart of water). Before removing gloves or handling anything else that HF on the gloves might be transferred to, carefully dip one hand at a time into the Epsom salts solution for a few seconds. Do not put the hand any deeper than necessary to bring the solution level to within 1-2 inches of the glove cuff. The Epsom salts solution will be prepared each day prior to beginning work with HF, and disposed at the end of the day down the laboratory sink.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled.

5.1.3. Latex, nitrile, and vinyl gloves provide adequate protection against the chemicals and reagents typically used during this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.1.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.6. Mercury is a highly toxic element that must be handled with care. Mercury vapor is toxic, so all work must be done in a functioning fume hood and vapors must be vented into a hood. If any volume of mercury reagent or liquid mercury is spilled, the Emergency Response Team must be activated, code yellow, for spill clean-up.

5.1.7. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.8. Do not look directly into the beam of the mercury lamp. The UV light from the lamp is harmful to the eyes.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydroxylamine Sulfate	Corrosive	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
			occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (1)	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Hydrofluoric Acid (1)	Poison Corrosive Dehydrator	3 PPM - TWA	Corrosive to the skin and eyes. Contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. Severely corrosive to the respiratory tract. Permanent eye damage may occur. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

EQUIPMENT

5.3. Preventative and routine maintenance is described in the “Schedule of Routine Maintenance” in the QAM Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

5.4. An autoclave capable of obtaining conditions of 15 psi at 120°C for 15 minutes. The temperature of the autoclave is verified daily using an "LASCAR" , or equivalent, temperature log device. In order to achieve a minimum 15 minutes at 120°C, the autoclave is set for a run duration of 25 minutes.

5.5. Automated mercury analyzers. Leeman PS20011 and HydraAA (or equivalent) both with autosamplers and WinHg Runner 1.5 software version CT Rev. 0.286 (or equivalent).

5.6. Computer with a printer.

5.7. Top-loading balance capable of accurately weighing 0.01 g.

5.8. 14 mL polystyrene test tubes for the autosampler.

5.9. 50 mL centrifuge tubes.

5.10. Nitrogen gas supply.

5.11. Pump windings – dimensions per instrument manufacturer requirements.

5.12. Bottle-top dispenser: re-pipetters.

5.13. Volumetric adjustable air displacement pipets.

5.14. Class A volumetric flasks.

5.15. pH indicator strips (pH range 0-14).

6. REAGENTS AND STANDARDS

6.1. Reagent water is produced by a Millipore nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Reagent water is checked on a daily basis as per SOP WS-QA-0014, (Monitoring Reagent Grade Laboratory Water).

6.2. Nitric acid (HNO_3), concentrated, analytical reagent grade.

6.3. Sulfuric acid (H_2SO_4), concentrated, analytical reagent grade.

6.4. Potassium permanganate, 5% solution (w/v): Dissolve 5.0 g of potassium permanganate for each 100 mL of reagent water.

6.5. Potassium persulfate, 5% solution (w/v): Dissolve 5.0 g of potassium persulfate for each 100 mL of reagent water.

6.6. Stannous chloride solution: Add 25 g of stannous chloride and 15 mL concentrated HCl to 250 mL of reagent water. This mixture is a suspension and should appear cloudy. This solution must be made every 12 hours.

6.7. Sodium chloride-hydroxylamine sulfate solution: Add 12.0 g of sodium chloride and 12.0 g of hydroxylamine sulfate for each 100 mL of reagent water.

6.8. All standards must be stored in polyethylene or polypropylene bottles.

6.9. 10 ppm mercury standards: Two separate stock standards purchased from separate manufacturers or different lots from the same manufacturer. One standard is used to make the ICV standard and the other for instrument calibration, CCVs, LCSs, and MS/SDs. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year from opening, and must be replaced sooner if verification from an independent source indicates a problem.

Note: Upon receipt of any stock standard, the "Certificate of Analysis" should be immediately filed in the proper location labeled with the receipt date. The receipt date and date the container is opened must be labeled on the container. Also write the new expiration date on the bottle which is the manufacturer's expiration date or one year after opening the stock standard.

6.10. 0.1 ppm working mercury standards (for each stock): add 0.5 mL of stock 10 ppm source standard and 1 mL HNO_3 to a 50 mL volumetric flask and dilute to volume with reagent water. These standards must be made daily.

7. SAMPLE COLLECTION, PRESERVATION AND STORAGE

7.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis.

7.2. Aqueous samples must be preserved with nitric acid to a pH of <2 at the time of sampling and may be stored in either plastic or glass. Refrigeration is not required. Preservation **must** be verified prior to analysis

8. QUALITY CONTROL

8.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be analyzed within the same time frame. A method blank (MB), laboratory control sample (LCS) or laboratory control sample duplicate (LCSD) are distilled and analyzed as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. An analysis batch must include all QC samples, however they do not

contribute to the maximum of 20 samples (see policy WS-PQA-003 (Quality Control Program) for more details).

8.2. One method blank must be processed and reported for every 20 samples. If a method blank exceeds +/- the reporting limit, then the samples must be re-extracted. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo (see policy WS-PQA-003(Quality Control Program) for further details).

8.3. A laboratory control sample (LCS) must be processed and reported for every 20 samples prepared and analyzed. A LCS consists of reagent water spiked with the analyte of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-extracted. One exception is when a LCS exhibits a high recovery, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo (see policy WS-PQA-003 (Quality Control Program) for further details).

8.4. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD pairs are aliquots of a selected field sample spiked with a known concentration of the analyte of interest. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and/or the MS/MSD may be required after evaluation and review.

Note: For air train samples the MS/MSD is typically performed on one Back Half (HNO_3/H_2O_2) fraction.

8.4.1. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for sample/sample duplicate nor MS/MSD analysis.

8.4.2. A laboratory control sample (LCS or LCS/LCSD) may be substituted when insufficient volume is provided to process a sample/sample duplicate or MS/MSD pair if required by program or client. The LCS and LCSD are evaluated independently for acceptance (see policy QA-003-SAC (Quality Control Program) for further details.

8.5. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard immediately upon completion of instrument calibration. This standard must be at a concentration different from that used to calibrate the instrument and different from the CCV standard. The ICV must fall within +/- 10% of the true value of the standard solution. An ICB prepared the same as

the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit from zero. If either the ICV or ICB fail to meet acceptance criteria the analysis must be terminated, the problem corrected, and the instrument re-calibrated.

8.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. Ten samples include any analysis that registers with a result, even if not used. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument re-calibrated, the calibration verified, and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action includes re-preparation of the associated samples.

9. CALIBRATION

9.1. All air displacement pipettes must be calibrated over their range of use at least monthly. If the analyst suspects a spiking volume problem, calibration may be required more often. See SOP WS-QA-0004 for pipettor calibration verification procedures.

9.2. All volumetric digestion vessels must be calibrated according to the frequency and procedures outlined in SOP WS-QA-0004. Prior to removing digestion vessels from the vendors box, verify that the box has a “TestAmerica Passed QC” sticker affixed to the outside. If there is not a sticker affixed, contact QA and have the lot checked before use..

9.3. All balances must be calibrated daily before use according to the procedures outlined in SOP WS-QA-0041.

9.4. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

9.5. Instrument calibration must be performed daily (once every 24 hours) or each time the instrument is off or non-operational for more than two hours.

9.6. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility

specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

9.7. Calibration standards must be processed through the preparation procedure as described in Section 11.9. Calibration standards must be prepared daily.

9.8. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a calibration blank. One standard must be at the reporting limit. Analyze standards in ascending order beginning with the blank

9.8.1. Calibration standard concentrations and the volume of 0.1 ppm working standard required to make 30 mL of standard:

0.1 ppm Hg Volume (mL)	0.06	0.15	0.3	1.5	3.0
Final Concentration (µg/L)	0.2	0.5	1.0	5.0	10.0

9.8.2. 30 g of 2% HNO_3 is added to each digestion vessel. An adjustable volumetric pipette is used to remove a volume of 2% HNO_3 corresponding to the spiking volume for that standard. The standard is then spiked with the appropriate volume of 0.1 ppm Hg standard.

9.9. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and re-calibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

9.10. The concentration of the ICV (section 9.5) is 2.0 ppb made by diluting 0.6 mL of the 0.1 ppm alternate source intermediate to 30 mL total volume with 2% HNO_3. See Section 10.4.2 for the spiking procedure.

9.11. The 5.0 ppb CCV is the same as the calibration standard.

9.12. The autoclave is monitored daily to assure samples are digested for the proper time interval at the appropriate temperature. An "ERTCO" temperature logging device is put in the autoclave and undergoes the same program as the samples. This device can be connected to a computer and a program generates a graph of time vs temperature. These are submitted with the raw data.

10. PROCEDURE

10.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

10.2. All preparation procedures must be carried out in a properly functioning hood.

10.3. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

10.4. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed. Double-check the sample bottle IDs, once prior to pouring them into the digestion vessels and again before they are returned to the sample cart.

10.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludge and tissues (animal/plant) are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge-like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

10.6. **Always** read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

10.7. The following procedure must be followed for all aqueous sample preparations:

10.7.1. Use a small aliquot of each sample and measure the pH with pH indicator strips. If the bottles have an “X” on the tops, they have already been tested for other tests and do not require this step.

10.7.1.1. For samples with pH<2 put an “X” on top of the bottle indicating the samples were properly preserved. For these samples, record pH<2 on the preparation log.

10.7.1.2. For samples that test pH>2, notify the project manager immediately so they can contact the client. If the client requests the sample be preserved by the laboratory, add HNO_3 in 1.0 mL aliquots until the sample remains at pH<2 for at least 10 minutes. Let the sample stand for 24 hours and re-test the pH prior to preparation. As long as the

pH<2, the sample can then be digested. A non-conformance memo must be created with an accounting of the anomalous event and the corrective action. A "Sample Preservation Log" must also be completed and filed with the raw data. Record "AF" for "Anomaly Filed" on the digestion log. Put an "X" on top of the bottle indicating the samples were properly preserved.

10.8. Sample Preparation:

10.8.1. All calibration and QC standards must be prepared in the same manner as the samples and be processed through all steps of the preparation procedure.

10.8.2. Transfer 30 mL of **well-mixed sample** to a 50 mL graduated centrifuge tube. The MS/SD sample should be shaken between pouring the separate aliquots.

10.8.2.1. For Air Train samples see SOP WS-IP-0007 for sample preparation procedures. For EPA Method 29, EPA Method 0060, or CARB 436 preparations on Air Trains:

10.8.2.1.1. Transfer 30mL of the $KMnO_4$ filtrate to a 50mL graduated centrifuge tube. (**WARNING:**: Front Half contains hydrofluoric acid.).

10.8.2.1.2. Transfer 30mL of the HCl filtrate into a 50mL graduated centrifuge tube.

10.8.2.1.3. Pipette 3mL of the Back Half Nitric Peroxide filtrate into a 50mL graducated centrifuge tube.

10.8.2.1.4. Pipette 3mL of the condensate into a 50mL graduatted centrifuge tube.

10.8.3. TCLP extracts are diluted 5X and STLC extracts diluted 10X prior to digestion. LCS and MS/SD spiking levels are 1.0 ppb times the dilution factor.

10.8.4. For each method blank and LCS required, weigh out 30 g of water. For the LCS, remove 0.3 mL with an adjustable volumetric pipette. Add 0.3 mL of the 0.1 ppm Hg intermediate used to make the calibration standards. The LCS mercury concentration is 1.0 ppb.

Note: For EPA Method 29, 0060 or CARB 36 samples add 27 mL of deionized nanopure H_2O to the Back Half (HNO_3/H_2O_2) and condensate.

10.8.5. For each MS/SD, spike the samples directly with 0.3 mL of the 0.1 ppm Hg intermediate used to make the calibration standards. The MS/SD mercury concentration is 1.0 ppb.

10.8.5.1. EPA Method 29 and EPA Method 0060: Add 27mL of DI Nanopure water to the Back Half Nitric Peroxide and Condensate.

10.8.6. Add 2.0 mL of concentrated H_2SO_4 and 1.0 mL of concentrated HNO_3, to all samples and standards, mixing after each addition.

10.8.7. Add 5.0 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If additional Potassium Permanganate needs to be added to one of the samples, it also need to be added to all other samples in the batch and the QC samples (MB, LCS, MS, MSD and the curve). If after the addition of up to 5 mL additional permanganate the color does not persist, sample dilution may be required.

Note: When reporting analyses, the addition of excess reagent must be addressed through mathematical correction of the results to account for the resultant dilution effect.

10.8.8. Add 2.5 mL of potassium persulfate solution.

10.8.9. Autoclave for 25 minutes at 120 °C and 15 lbs (to maintain temperature for 15 minutes).

10.8.9.1. Allow the autoclave to cool to less than 80°C where there is no psi of pressure remaining.

<u>Warning:</u> Caution must be used when opening the door of the autoclave as superheated steam may be present and can cause significant burns.

10.8.10. Allow all of the samples to cool to room temperature. Check that the volume is 30 mL. If volume is <30 mL, add reagent H_2O to bring volume to 30 mL. If volume is >30 mL, record the volume.

10.9. Sample Analysis:

10.9.1. When ready to begin analysis, add 2mL of sodium chloride-hydroxylamine sulfate solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Mercury analysis is run using a Leeman PS200II or Hydra AA automated mercury analyzer. Results are reported as ug/L Hg.

10.9.1.1. Make certain the lamp is on and the pump windings are working properly. Replace any pump windings that do not exhibit a smooth flow of liquid or look stretched or flattened.

<u>Warning:</u> Do not look directly into the beam of the mercury lamp. The UV light from the lamp is harmful to the eyes.

10.9.1.2. Load the samples into the automated sampler racks according to the sample lists entered into the computer. Do a final check once all samples have been loaded on to the racks to assure proper sequencing.

10.9.1.3. Stannous chloride is automatically added to the samples by the instrument via the peristaltic pump and mixed with the sample in a liquid-gas separator. This must be freshly prepared every 12 hours.

10.9.2. Dilute and reanalyze all samples that exceed the 10 ppb linear range of the calibration.

10.9.3. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

10.9.4. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.9.5. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

10.10. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data is reviewed periodically throughout the run.

11. CALCULATIONS/DATA REDUCTION

11.1. ICV/CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found_{(ICV\,/\,CCV)}}{True_{(ICV\,/\,CCV)}}\right)$$

11.2. Matrix spike recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result
SA = Spike Added

11.3. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

11.4. The final concentration for an aqueous sample is calculated as follows:

$$\mu g/L = C \times D$$

Where:
C = Concentration ($\mu g/L$) from instrument readout
D = Instrument dilution factor

11.5. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found_{(LCS)}}{True_{(LCS)}}\right)$$

11.6. Appropriate factors must be applied to sample values if dilutions are performed.

11.7. Sample results should be reported with up to three significant figures in accordance with the TestAmerica Sacramento significant figure policy (see QA-004-SAC, Rounding and Significant Figures).

12. METHOD PERFORMANCE

12.1. Method Detection Limit

12.2. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure

for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-006 and policy S-Q-003.

12.3. Initial Demonstration

Each analyst must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

12.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration standard.

12.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the historical acceptance criteria.

12.3.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.4. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14. WASTE MANAGEMENT

14.1. The following waste streams are produced when this method is performed.

14.1.1. Unused acidic digestate from the preparation process and remaining in the plastic tubes on the instrument. This acidic liquid is consolidated into a plastic LLE drum. When the drum is full or after no more than 75 days, move this drum to the main waste area for shipment

14.1.2. Aqueous acidic waste from the auto-analyzer. This is collected in a 1-gallon

carboy. When the carboy is full, or after no more than one year, consolidate it into a plastic LLE drum for shipment.

14.1.3. Contaminated plastic vials from digestion and analysis. Pour any excess/unused sample from the vial into the aqueous acidic waste drum. Put the plastic vial into the contaminated lab trash. Collect all of the contaminated vials in a plastic bag, and move it to the waste collection area for shipment.

15. REFERENCES/CROSS REFERENCES

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

15.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.1.

15.3. WS-PQA-003, Quality Control Program.

15.4. WS-PQA-004, Rounding and Significant Figures.

15.5. WS-QA-006, Method Detection Limits and Instrument Detection Limits.

15.6. WS-QA-023, Nonconformance and Corrective Action System.

15.7. WS-IP-0007, Determination of Metals Emissions From Stationary Sources

16. METHOD MODIFICATIONS

16.1. Modification from Method 7470A and 245.1.

16.1.1. The method has been modified for use with Leeman automated analyzers. The samples are prepared to a total volume of 30 mL versus the 100 mL volume specified in both methods. The addition of all reagents is modified relative to this decrease in volume. Stannous chloride in HCl is used in place of Stannous sulfate in H_2SO_4 (per instrument instructions). The analytical method is automated, whereas both methods are written for manual CVAA analysis.

16.1.2. STLC leachate analysis has been added to the methods.

16.1.3. The five point curve used by TestAmerica Sacramento has a 0.2 ppb Hg standard to accommodate analysis of one standard at our 0.2 ppm reporting limit. Both reference methods start with a 0.5 ppb Hg standard and include a 2.0 ppb Hg standard. We do not run the 2.0 ppb Hg standard.

16.1.4. This method has been modified for use with the autoclave as an alternate digestion procedure (section 10.8.9).

17. ATTACHMENTS

17.1. Appendix I - MSA Guidance

17.2. Appendix II – Autoclave Usage Letter from RCRA National Inorganic Program

17.3. Appendix III - Contamination Control Guidelines

18. REVISION HISTORY

18.1. WS-MT-0005, Revision 5.5, Effective 07/15/2014

18.1.1. Method modification updated to include autoclave as an alternate digestion procedure.

18.1.2. Inserted autoclave usage letter from RCRA National Inorganic Program identified as Appendix II.

18.2. WS-MT-0005, Revision 5.4, Effective 06/22/2012.

18.2.1. Updated Section 10.8.7 to address procedure for adding extra Potassium Permanganate to samples.

18.2.2. Editorial changes.

18.3. WS-MT-0005, Revision 5.3, Effective 12/23/2011.

18.3.1. Updated Section 5 to address safety precautions to use with HF acid.

18.3.2. Added Section 6.1 addressing preventative maintenance statement.

18.3.3. Updated Section 6.2 addressing autoclave conditions and monitoring.

18.3.4. Updated Section 6.3 addressing instrumentation and software

18.3.5. Editorial changes.

18.4. WS-MT-0005, Revision 5.2, Effective 02/17/2010

18.4.1. Editorial changes.

18.4.2. Revised note following Section 7.9 to include expiration date on label affixed to standard bottle.

18.4.3. Revised Section 7.1 to read “Reagent water is checked on a daily basis as per SOP WS-QA-0014, (Monitoring Reagent Grade Laboratory Water).

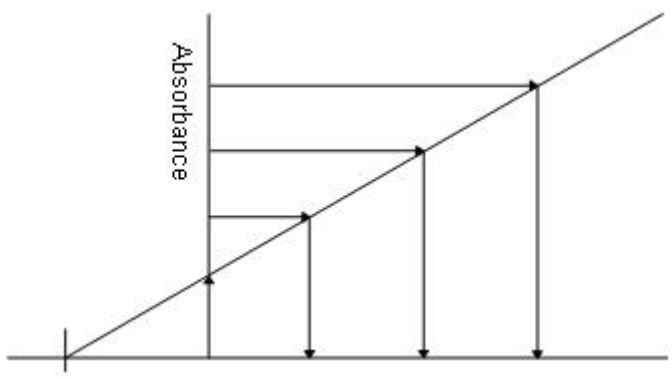
18.4.4. Revised Section 10 to include parameters for calibrating pipettors, balances, and digestion vessels.

APPENDIX I. MSA GUIDANCE
Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX II
AUTOCLAVE USAGE LETTER

Ms. Debra K. White
Principal Inorganic Scientist
Quanterra Incorporated
4955 Yarrow Street
Arvada, CO 80002

Dear Deb,

Thank you for your letters of July 10, 1995 and September 25, 1995, seeking clarification on several issues regarding RCRA testing and analysis. As the Office of Solid Waste updates SW-846 methods, we will take every opportunity to incorporate your comments and suggestions. Our goal is to remove overly restrictive language from SW-846 methods, which does not effect method performance and to continue to move toward truly performance based methods.

Your first request is for a clarification regarding the acceptance of the autoclave to prepare aqueous samples for mercury analysis under EPA SW-846 Method 7470A. The heating device in Section 4.10 of the Method 7470A specifies "Hot plate or equivalent." An autoclave would classify as an equivalent heating device and should give equivalent results to the hot plate/water bath protocol called for in Sections 7.1 and 7.2 of Method 7470A. Method 245.1 CLP-M is analogous to Method 7470A and allows the autoclave option for sample preparation.

Your second request is for a clarification regarding scaling of sample size for RCRA analysis. In particular, you request processing 50 mL aliquots for aqueous metal digestion rather than the 100 mL sample size specified in the methods. This should not present any problems for pure aqueous samples (no solids) because of their homogeneous nature. As a "representative sample" can be assured, scaling causes no loss of precision or accuracy in the analysis. Solid samples on the other hand are frequently heterogeneous in nature and changing sample size presents a big problem in obtaining a "representative sample" and should not be allowed without proper sample preparation (ie. crushing, grinding, mixing, and splitting).

I hope that this information is helpful to your analytical program. If you have any questions, please feel free to call me at (202) 260-4778.

Sincerely,

Oliver M. Fordham, Jr.

National Inorganic Program
Manager for RCRA

APPENDIX III. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause-cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: Extraction of Semivolatile Organic Compounds for Analysis by Method 8270C, Based on SW-846 3500 Series and 3600 Series, and PAH-SIM by Internal Standard and Isotope Dilution Procedures

[Method 8270C]

Approvals (Signature/Date):

Liz Koston — 8/14/14 — Date Organics Extractions Manager	Joe Schairer — 8/14/14 — Date Health & Safety Manager / Coordinator
Lisa Stafford — 8/14/14 — Date Quality Assurance Manager	Robert Hrabak — 8/18/14 — Date Laboratory Manager

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1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and concentration) of semivolatile organic analytes in aqueous, TCLP leachate, soil and waste matrices for analysis by Gas Chromatography/Mass Spectrometry (GC/MS). The procedures are based on SW-846 and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA).

1.1. Extraction procedures for the following determinative methods are covered: 8270C and 8270C (modified). 8270C (modified) encompasses analysis for 1,4-Dioxane, PAHs by Selected Ion Monitoring (PAH-SIM), and PAHs by Selected Ion Monitoring-Isotope Dilution (PAH-SIM-ID). The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.2. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

1.3. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction: A measured volume of sample is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.1.1. This method is subdivided into two extraction methods: a 1000mL nominal sample volume extracted in 2L separatory funnels (henceforth referred to as a 1L separatory funnel extraction in this SOP), and a 250mL sample volume extracted in a 500mL separatory funnels (henceforth referred to as a Large Volume Injection/Reduced Volume Initiative or LVI/RVE in this SOP).

2.2. Microwave Extraction: A measured weight of sample, typically 15g (10 g for biologics), is extracted with methylene chloride in a microwave extraction apparatus.

2.3. Sonication Extraction: A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent-extracted three times using an ultrasonic horn.

2.4. Soxhlet Extraction: A measured weight of sample, typically 15 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent for 16-24 hours.

2.5. Concentration: Procedures are presented for drying and concentrating the extracts to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3. To prevent analyte loss, extracts which are not immediately concentrated by KD must be protected from light during storage. Extracts may be either refrigerated or stored in an opaque container.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile gloves must be used. Latex gloves may be used for methanol.

5.1.2. The use of solvent dispensing pumps and solvent squeeze bottles are high risk activities, and a face shield must be worn over safety glasses or goggles while using a dispensing pump.

5.1.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. When Soxhlet extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.1.5. Laboratory procedures such as microextraction of water samples in VOA vials, repetitive use of pipettes, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.6. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the process may be performed behind a closed fume hood sash. After each of the three extraction cycles, ensure that the stoppers are removed from the top of the separatory funnels.

5.1.7. The use of ultrasonic sonicator systems to extract soil samples creates a hazard to hearing. Sonication must be performed inside of a fume hood, with all sashes on the hood closed. The operator must wear hearing protection while the sonicators are functioning.

5.1.8. Assembly and disassembly of glassware, including stopcocks and stoppers, creates a risk of breakage and cuts. All staff members shall wear Kevlar® or similar cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.9. The use of vacuum systems during rotovap concentration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Refer to SOP WS-OP-0011 for details of glassware cleaning. Kilned glassware does not require solvent rinsing before use.

WARNING: Heat resistant gloves must be used when handling kilned glassware.

6.2. Equipment and supplies for extraction procedures

6.2.1. Separatory funnel, 2L (Teflon or Glass)

6.2.2. Separatory funnel, 500mL (Teflon or Glass)

6.2.3. Separatory funnel rotator with timer

6.2.4. Balance: >1400 g capacity, accurate ± 0.1 g

6.2.5. pH indicator paper, wide-range: covers extraction pH

6.2.6. Graduated cylinder: 1 liter. (Other sizes may be used)

6.2.7. Erlenmeyer flask, beaker, or French Square jar: 125 & 300 mL (other sizes optional)

6.2.8. Solvent dispenser pump or 100 mL graduated cylinder

6.2.9. Continuous liquid/liquid extractor

6.2.10. Round or flat bottom flask: 250, 500 mL or 1 L

6.2.11. Boiling chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).

6.2.12. Cooling condensers

6.2.13. Heating mantle: Rheostat controlled, with timer

6.2.14. Beakers: 250 & 400 mL, graduated

6.2.15. Balance: >100 g capacity, accurate to ± 0.01g

6.2.16. Soxhlet extractor

6.2.17. Sonicator (at least 300 watts), with sonicator horn, 3/4 inch. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2.18. Kuderna-Danish (KD) apparatus: 500 mL, with 10mL concentrator tube, and 3-ball macro Snyder Column

6.2.19. Locally assembled solvent vapor condensing/recovery system for use with 6.2.18 above.

6.2.20. Water bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.

6.2.21. Nitrogen blowdown apparatus.

6.2.22. Culture tubes: 10 mL, 16 mm x 100 mm

6.2.23. Autosampler vials and caps

6.2.24. Gastight syringe 1 mL or 0.5 mL that is manufactured to a certified volume delivery tolerance of ± 0.01 mL

6.2.25. Glass wool
To ensure glass wool is free of contaminants, it is necessary to soxhlet rinse glass wool in methylene chloride for 18 hours.

6.2.26. Glass funnel: 75 X 75 mm

6.2.27. Disposable pipets

6.2.28. Aluminum foil

6.2.29. Paper towels

6.2.30. 60 mL VOA vial w/ teflon lined caps

6.2.31. Pressure rotor and pressure reactor

6.2.32. Extraction vessel and extraction vessel cover

6.2.33. Glass inserts

6.2.34. Microwave – Milestone Ethos EX

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1.1. Sodium hydroxide (NaOH) solution, 10 N, reagent grade

7.1.2. Sulfuric acid (H_2SO_4), concentrated; reagent grade

7.1.3. Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.

WARNING: Always add concentrated acids or bases to water, never add water to the concentrated acid or base solution. Due to the reaction between acid and water, add the acid slowly to water to prevent breaking of the mixing vessel

7.1.4. Organic free reagent water (deionized water). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks, in accordance with SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water."

7.1.5. Sodium sulfate (Na_2SO_4), granular, anhydrous, reagent grade: Purify by heating at 400°C for a minimum of two hours.

7.1.6. Extraction/exchange solvents: Methylene chloride, pesticide quality or equivalent, and 1:1 methylene chloride:acetone.

7.1.7. Acetone: Used for cleaning

7.1.8. Nitrogen, reagent grade

7.2. Reagents for Cleanup Procedures

7.2.1. Oasis HLB Extraction Cartridge: 500 mg pre-packed in 6mL cartridges with polyethylene frits (Waters part # 186000115, or equivalent).

7.2.2. Activated Silica Gel: Activate for at least 1 hour at 190°C (± 5°C). Store at 130-200°C in a covered glass container.

7.3. Standards

7.3.1. Source Standards

Source standards are purchased as certified solutions or prepared from neat materials. Semivolatile source standards are stored in a refrigerator at ≤ 6ºC, unless otherwise specified by the manufacturer. All source standards must be protected from light.

7.3.1.1. Source standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Standards must be allowed to come to room temperature before use.

7.3.1.2. Expired standards must be rotated out of the lab to the Hazardous Waste storage area.

7.3.2. Surrogate Spiking Standards/Isotope Dilution Internal Standards

Prepare or purchase surrogate spiking standards as described in Tables 4 and 5. Surrogate spiking standards are prepared as dilutions of the source standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards are given a one year expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the Matrix Spike (MS), Matrix Spike Duplicate (MSD), and the Laboratory Control Sample (LCS). Prepare MS/MSD/LCS spiking standards as described in Table 4. Spiking standards are purchased or prepared as dilutions of the source standards. Spiking solutions must be refrigerated and protected from light. The standards are given a one year expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4 ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2. For TCLP leachates, extraction is initiated within seven days from when the leaching procedure is initiated.

8.3.3. Analysis of the extracts is completed within forty days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a MB, an LCS, and a MS/MSD. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy WS-PQA-003 for further definition of the batch.

9.2. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented using Clouseau.

9.3. Method Blank (MB)

A MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.3.1. Aqueous MB use 1000mL (1L separatory funnel extraction) or 250mL (LVI/RVE) of organic free reagent water (deionized water) spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.2. Solid MB use 30g of kilned sodium sulfate for 3550B/C and 15g ottowa sand

for 3546 spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.3. TCLP MB use 200 mL of leachate fluid spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.4. Waste MB use methylene chloride spiked with the surrogates. The MB goes through the entire analytical procedure, including any cleanup steps.

9.4. Laboratory Control Sample (LCS)

LCS is a well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.4.1. The LCS is made up in the same way as the method blank (see Sections 9.3.1 - 9.3.4) but is spiked with the LCS standard and the surrogate.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS is an environmental sample to which known concentrations of target analytes have been added. A MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. The MS and MSD are spiked with the LCS standard and the surrogate.

9.7. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.9. TestAmerica Sacramento QC Program

Further details of QC and corrective action guidelines are presented in the TestAmerica Sacramento QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. On the day of use, measure either 0.5 mL or 1.0 mL of solvent into an autovial using a 1mL or 0.5mL gastight syringe that is calibrated in accordance with SOP WS-QA-0004. Seal the autovial – this is the 'check' or 'reference' vial. On the day of use, measure 10.00mL of solvent into a culture tube using a 5.00mL gastight syringe or sereological pipet that is calibrated in accordance with SOP-WS-QA-0004. Record the autovial or culture tube lot number, solvent, gastight syringe or sereological pipet ID, date, and initial in a controlled logbook. The autovials containing the sample extracts are then compared against the "check" vial to ensure that the final volume is consistently 1.0 ± 0.03 mL or 0.5 ± 0.015 mL depending on the analysis.

10.2. On a daily basis, calibrate any auto-pipettors to be used in accordance with SOP WS-QA-0004.

10.3. On the day of use, calibrate any balances to be used in accordance with SOP WS-QA-0041.

10.4. Prior to use, ensure any containers used for quantitative steps (e.g final volume) are calibrated with WS-QA-0004.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and LCS spiking standards from refrigerator and allow standards to warm to room temperature.

11.2.2. If the sample bottle contains a layer of sediment greater than approximately ½" in depth, contact the department manager and the project manager to determine how to proceed with the sample extraction. Options may include the following:

a) Decanting and extracting only the liquid fraction in a separatory funnel before adding the surrogate and/or spike solutions.
b) Adding the surrogate and/or spike solutions to the sample with the sediment still in the original sample container.
c) Processing the aqueous portion as described in a) along with creating a new sample id for the soil matrix. The sample bottle would then be rinsed with 60 mL of DCM, spiked with an appropriate volume of surrogate solution and shaken for 3 minutes. The addition of 60 mL of DCM will be repeated 2 more times and all solvents combined to create a second extract for the sample that is to be added to the extraction batch of water samples

NOTE: In the case of option "b", after adding the appropriate surrogates/spikes, the sample would be gently swirled for mixing purposes and then gently poured into the separatory funnel, thus minimizing the amount of sediment transferred to the extraction vessel during the process. The original sample container would then be rinsed with 60 mL of DCM. This DCM rinseate would then be poured into the extraction vessel.

11.2.3. Measure the initial sample pH with wide-range pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against the leachate log and note on the benchsheet if there is any discrepancy.

11.2.4. The normal sample volume is 1 liter for 1L separatory funnel extraction and 250mL for LVI/RVE. Other sample volumes may be used to obtain specific reporting limits, or accommodate dirty samples and reduced sample volumes, diluted to 1 liter or 250mL with organic free reagent water (deionized water).

11.2.5. Obtain sample weight(s) to ± 0.1 g. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. This may be done in two ways depending if a dilution is necessary.

11.2.5.1. For samples not requiring a dilution weigh the gross container on a tared balance and after extraction has been initiated weigh the

tared container on a tared balance and use the difference as the sample weight.

11.2.5.2. For samples requiring a dilution, subsample the desired aliquot amount in accordance with SOP-QA-0018 into a tared container. Dilute to 1L nominal for 1L separatory funnel extraction and 250mL nominal for LVI/RVE with organic free reagent water (deionized water).

11.2.5.3. For TCLP samples, use 200mL of leachate and dilute to 1L in organic free reagant water.

Note: *Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.*

11.2.6. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.2.7. For a TCLP batch prepare a MB as specified in section 9 of this SOP. In addition, prepare a TCLP method blank by measuring 200 mL of buffer solution used in the leaching procedure and dilute to 1L nominal using the method described in 11.2.4. No leachate LCS is required by this method.

11.2.8. After samples have warmed to room temperature, add the surrogate and spike standard to the necessary samples as described in section 9 and Table 11 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer part of the sample to the separatory funnel and then add the spike to the bottle.

1L Separatory Funnel Extraction Spiking Volumes				
Test	**Surrogate Solution**		**Matrix Spike Solution**	
	Volume	**Name**	**Volume**	**Name**
8270C	0.5mL	8270Surrogate	1.0mL	8270 Spike
1,4-Dioxane	50µL	8270 Surrogate	50µL	1,4-Dioxane
PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike Mix
PAH-SIM ID	0.10mL	PAH-IS	0.05mL	PAH-ID N.S.

LVI/RVE Spiking Volumes				
Test	**Surrogate Solution**		**Matrix Spike Solution**	
	Volume	**Name**	**Volume**	**Name**
8270C	0.12mL	8270 Surrogate	0.25mL	8270 Spike
PAH-SIM	0.12mL	PAH Surrogate	0.12mL	PAH Spike Mix

Note: Contents of each solution are described in the Tables in Section 17

11.2.9. For regular 8270C samples, adjust sample pH to between 1 and 2. Use the minimum amount of 1:1 H2SO4 necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper.

Note: *Samples for PAH-SIM and 1,4-Dioxane analysis are extracted at neutral pH (pH 5 – pH 9). These samples should not require pH adjustment. If samples do require pH adjustment, use the minimum amount of 1:1 H_2SO_4 or 10N NaOH as necessary.*

11.2.10. Record adjusted pH, spiking volumes standard numbers, and reagent lots on the benchsheet.

11.2.11. Mix well, or if the sample contains sediment, decant before transfer. Transfer the sample to the separatory funnel. Rinse the sample bottle with 60mL methylene chloride for 1L separatory funnel extraction and 30mL for LVI/RVE and transfer to the 2L glass or Teflon separatory funnel for 1L separatory funnel extraction and 250mL glass or Teflon separatory funnel for RVE/LVI.

11.2.12. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. As described in 5.1.6, Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle.

WARNING: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.13. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (at least 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may

include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. One ideal method is to swirl after resting for the required amount of time, wait approximately a minute, and then drain the extract without collecting the emulsion. One the third and final time, add a squirt of DCM to identify where the layer of DCM and water is separated and collect only the DCM. Alternatively, on the final pour collect the emulsion and use a second filtration step at KD with glass wool and pipet off excess water.

***Note:** 15 – 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 40 mL from the first shake and still be acceptable for 1L separatory funnel extraction. Subsequent shakes should recover at least 50 mL of solvent for 1L separatory funnel extractions. Apply the same ratios for LVI/RVE*

11.2.14. Fill a funnel with anhydrous kilned sodium sulfate (10-15g for LVI/RVE, nearly a full funnel for 1L separatory funnel extraction). The funnel can be plugged with glass wool to hold the sodium sulfate. Rinse the funnel with a small amount of methylene chloride, and discard the rinsate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

11.2.15. Repeat the extraction process two more times using fresh portions of solvent – 60 mL for 1L separatory funnel extraction and 30mL for LVI/RVE, combining the three solvent extracts in the collection container. Enusre to minimize the amount of water collected. If extraction at a secondary pH is not required, proceed to Section 11.2.17.

11.2.16. If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to between 11 and 12 with a minimum amount of 10 N NaOH. Measure with pH paper and record the adjusted pH on the benchsheet. Serially extract with three 60mL portions of methylene chloride for 1L separatory funnel extraction and three 30 mL portions of methylene chloride for LVI/RVE, as outlined in Sections 11.2.12 to 11.2.15. Collect these three extracts in a separate bottle. Once again, minimize the amount of water collected.

11.2.17. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.18. Dispose the solvent-contaminated water remaining in the extractor into the

LLE waste drum. Waste methylene chloride goes to the solvent waste drum for recycling.

11.2.19. If the extract is not concentrated immediately, refrigerate it to protect from light. Alternatively, the extract bottle may be wrapped with foil to protect from light for short periods of time (i.e., overnight). Refer to Section 11.6 for concentration.

11.3. Method 3546, Solid Extraction by Microwave

11.3.1. Remove surrogate and matrix spiking solutions from refrigerator and allow standards to warm to room temperature.

11.3.2. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.3.3. Weigh 15g of sample (10g for biologics) ± 1.0g into an extraction vessel. Record the weight to the nearest 0.01g in the appropriate column on the benchsheet.

11.3.3.1. For visually dirty samples, it may be necessary to weigh the sample into a glass insert to be placed into an extraction vessel. Contact your supervisor to determine if this is the case.

11.3.4. Mix weighed sample with a spatula. If clay clumps or other sample aggregation is evident, add Ottawa sand (not to exceed 5g) as needed to facilitation disaggregation and mix as necessary.

11.3.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.3.6. Add the surrogate and spike standard to the necessary samples as described in section 9 and Table 11 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.3.7. Immediately add 25mL of DCM to extraction vessels. Note: Steps 11.3.6 - 11.3.7 should be performed rapidly to avoid loss of the more volatile extractables.

11.3.8. Add flared extraction vessel covers to each extraction vessel containing sample and QC aliquots.

11.3.8.1. It is important that each cover fits snugly to ensure a proper seal. The cover should not slide easily or loosely inside the extraction

vessel, but should require some finger pressure to insert firmly. A cover flaring tool should be used.

11.3.8.2. For the visually wettest sample, add the thermowell liner into the extraction vessel cover to create the representative sample that the ATC temperature sensor can be inserted into.

11.3.9. Place each extraction vessel into a pressure reactor. Screw on the pressure cap/safety lid. The pressure cap should be hand tightened until the sealing valve is flush with the top of the cap.

11.3.9.1. For the representative sample created in 11.3.8.2, add the protection foil and appropriate safety lid.

11.3.10. Place all the extraction vessels into the rotor so that the pressure-release valves are facing outside of the rotor on the outside ring and inside toward the center on the inside ring.

11.3.10.1. Place the rotor in the microwave oven and insert the ATC temperature sensor into the representative sample (11.3.8.2 and 11.3.9.1).

11.3.11. Close the microwave oven and start the appropriate extraction profile.

11.3.11.1. A 10 minute ramp from 25C to 100 C, hold for 25 minutes at 100C, followed by a 15 minute cooldown to 25C

11.3.12. After the extraction period and cooldown, pressure reactors should be at ambient temperature prior to removal from the rotor and opening.

11.3.13. Proceed to section 11.6 concentration

11.3.13.1. Discard any glass inserts. To clean extraction vessels and pressure cap/safety lid, use soap and water followed by a solvent rinse for. Use only solvent to rinse the probe. Allow to air dry prior to use.

11.4. Method 3550B and 3550C, Sonication

11.4.1. Remove surrogate and matrix spiking solutions from refrigerator and allow them to warm to room, temperature.

11.4.2. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.3. Weigh 30 g of sample ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass

jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0.1 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate for the method blank and LCS.

For PAH-SIM use 10g of sample instead of 30 g of sample.

11.4.4. Mix weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.5. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.4.6. Add the surrogate and spike standard to the necessary samples as described in section 9 and Table 13 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.4.7. Immediately add 100 mL of 1:1 methylene chloride:acetone for 8270C and methylene chloride only for PAH-SIM to the beaker.

Note: *Steps 11.5.5 - 11.5.7 should be performed rapidly to avoid loss of the more volatile extractables.*

11.4.8. Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pour beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horns with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

11.4.9. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.10. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: *Do not use Microtip probe.*

WARNING: Hearing protection is required when sonicating samples.

11.4.11. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.4.12. Place the prepared funnel on a collection apparatus (beaker or KD).

11.4.13. Decant and filter extracts through the prepared funnel into a clean beaker or KD.

11.4.14. Repeat the extraction two more times with additional 60-100 mL minimum portions of solvent each time. Decant and retain the extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

11.4.15. If the extract is not concentrated immediately, refrigerate it to protect from light. Alternatively, the extract bottle may be wrapped with foil to protect from light for short periods of time (i.e., overnight). Refer to Section 11.6 for concentration.

11.4.16. Sonicator Tuning.

11.4.16.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed.

11.5. Waste Dilution

11.5.1. This method is used for materials that are soluble in an organic solvent.

11.5.2. Remove surrogate and matrix spiking solutions (see Table 7) from refrigerator and allow to warm to room temperature

11.5.3. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest 0.01 g.

11.5.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.5.5. Add the surrogate and spike standard to the necessary samples as described in section 9 and Table 13 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking. Note, it may not be necessary to spike all analytes and comparing the analyst list in TALS to the analytes in each standard may be beneficial.

11.5.6. Compare the volume to that of the calibrated reference container, and carefully add methylene chloride to make the final volume of 10 mL. Ensure to record the lot of the container that extracts are brought to final volume in..

11.5.7. Add approximately 2g of sodium sulfate to the samples. Cap and shake for 2 minutes.

11.5.8. Aliquot 1.0 mL into a calibrated autosampler vials using a reference container.

Ensure to record the lot of the autosampler vial. Store the vial box in the refrigerator at 0-6°C in the SVOA instrument lab until ready for GC/MS

11.5.8.1. The excess in the culture tube is stored in the refrigerator at 0 - 6°C for a minimum of 30 days (can be used for additional re-analysis as needed) and disposed in accordance with Section 15.

11.6. Concentration

According to the type of analysis and any cleanup procedures needed, different final volumes may be required. Below are the "normal" final volumes for tests covered by this SOP:

Test	Aqueous	Solid
8270C	1.0 mL	1.0 mL
1,4-Dioxane	1.0 mL	NA
PAH-SIM (all except LVI/RVE)	1.0 mL	1.0 mL
PAH-SIM LVI/RVE	0.5mL	NA

11.6.1. Kuderna-Danish (KD) Method:

11.6.1.1. For aqueous samples, loosely plug the stem of a 75mm x 75mm glass funnel with glass wool and rinse well with DCM. For solid samples place a folded piece of Whatman #1 filter paper into the glass funnel.

11.6.1.2. For LVI/RVE samples, add approximately 10-15 g of anhydrous sodium sulfate to the funnel cup. For 1L separatory funnel extraction, add anhydrous sodium to nearly fill up the funnel cup. For solid samples, add approximately 5-10 g of anhydrous sodium sulfate to the funnel cup.

11.6.1.3. Pour the sample through the prepared glass funnel into the KD flask. For 8270 Aqueous, add the base portion then immediately add the acid portion. Add one or two clean boiling chips to the KD flask.

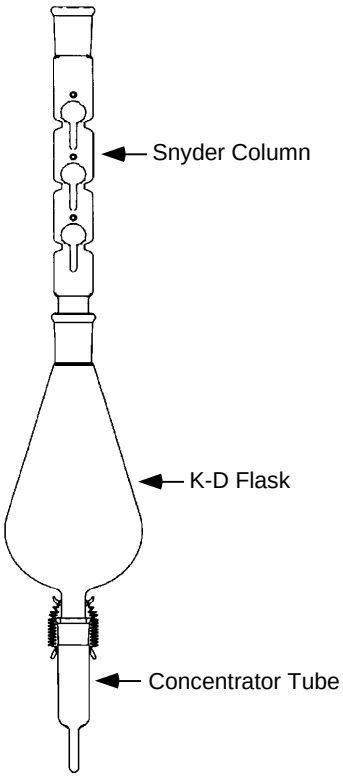
11.6.1.4. Rinse the top of the funnel as well as the sample container with DCM.

11.6.1.5. Prepare the three ball Snyder columns by rinsing them with DCM to ensure that the balls are not stuck and that the column will work properly.

WARNING: Use of cut-resistant gloves (Kevlar® or similar material) is required when assembling or disassembling glassware.

11.6.1.6. Assemble a Kuderna-Danish concentrator as shown in Figure 2 by attaching a 10 mL concentrator tube to the 500 mL KD flask. Transfer the sample to the KD flask. Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three-ball Snyder column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly).

Figure 2, KD Setup (See Section 11.8. for description)



11.6.1.7. Place the KD apparatus on a water bath (70-75ºC for all except 8270 solid at 80-85 ºC) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood. Attach the solvent vapor recovery device to the top of the Snyder column.

11.6.1.8. Concentrate to 5-15 mL. The Snyder column may be insulated if necessary to maintain the proper rate of distillation. For 8270 Aq, add a squirt DCM to the top of the snyder column at about 30mL.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

WARNING: Do not concentrate to dryness as an unsafe condition may be created.

11.6.1.9. Remove the KD apparatus from the water bath and allow it to cool and drain. For 8270 aqueous, rinse the inside of the joint with a minimum amount of DCM rotating the flask as necessary to ensure the rinsate covers the sides of the KD flask. For 8270 aqueous, repeat a second time.. If the level of the extract is above the level of the concentrator tube joint, add new boiling chips and continue to distill the solvent as necessary.

11.6.2. Nitrogen Evaporation to Final Concentration

Note: If the extracts are proceeding to clean-up, skip to Section 11.6.3 (Nitrogen Evaporation to Volume for Clean-up).

11.6.2.1. Evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

11.6.2.2. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1mL. Concentrate the solvent to below 1mL (below 0.5mL for PAH-SIM analysis) and quantitatively transfer the extract to the storage vial. .

11.6.2.2.1. For 8270 AQ, dunk the concentrator tube in water, heated to a maximum of 3.5 on the N-evap. Remove when the sovlent level is at about 700uL on the concentrator tube (~500uL of solvent).

11.6.2.3. Quantitatively transfer the extract to calibrated autosampler vials. Compare the volume to that of a calibrated reference, and carefully add solvent to raise the level to the proper amount. Ensure to record the lot of the container that extracts are brought to final volume in. The extract is now ready for instrumental analysis.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note: *The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.*

11.6.3. Nitrogen Evaporation to Volume for Clean-up

Note: If the extracts are not proceeding to clean-up, go to the previous Section (Nitrogen Evaporation to Final Concentration).

11.6.3.1. Transfer the entire extract to an evaporation tube. Rinse the concentrator tube with 1-2mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

11.6.3.2. Evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

11.6.3.3. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1mL. Concentrate the solvent to 1 – 1.2mL.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note: *As the entire sample will go through the clean-up column and be adjusted to an exact volume afterward, the extract volume at this step is not critical.*

11.6.3.4. Proceed to the appropriate Clean-up option in the following sections.

11.6.4. Nitrogen evaporation for final volume (Microextraction)

11.6.4.1. Aliquot 5mL and transfer to 8mL screw cap test tube.

11.6.4.2. Evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

11.6.4.3. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1mL. Concentrate the solvent to 1.0mL.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note*: As the entire sample will go through the clean-up column and be adjusted to an exact volume afterward, the extract volume at this step is not critical.*

11.7. Clean-up of 8270 extracts with HLB column cartridges

This pre-packed column may be used to trap co-extracted matrix contamination and yield cleaner 8270 extracts. This clean-up is recommended for 8270 extracts from soil and heavily contaminated water.

Refer to Section 11.9 for the HLB lot verification procedure.

Note: *Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assembly, Zymark Benchmate, or equivalent.*

11.7.1. Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.7.2. Place a valve liner (Supelco, P/N# 57059, disposable flow control valve liner, or equivalent) into each port of the vacuum manifold.

WARNING: Use of vacuum systems creates a significant risk of implosion. Thoroughly inspect all glassware and do not use any that has been chipped, rubbed, cracked, or marred in any fashion.

WARNING: Ensure that the exhaust line from the vacuum pump is secured well inside of a fume hood so that it cannot fall out of the hood.

11.7.3. Place one HLB cartridge into the vacuum manifold for each sample extracts.

11.7.4. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of DCM, then 5mL of MeOH.

11.7.5. During this step, adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min.

11.7.6. Add 2mL of MeOH, drain through the column but stop the flow when the MeOH reaches the top of the cartridge. Do not let the cartridge go dry.

11.7.7. Place a rack of clean, labeled 16 mL test tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.7.8. After the clean tubes are in place, vacuum to the manifold is restored, add the extract to the appropriate cartridge. This is the entire sample.

11.7.9. The extract concentrates are then eluted through the column with 10 mL of acetone and are collected into the 16 mL test tube or held in the rack inside the vacuum manifold.

11.7.10. Use nitrogen evaporation (Section 11.8.2) to concentrate the extract to approximately 1 mL. Add 5mL DCM and re-concentrate to 1.0mL (for a thorough removal of acetone).

11.7.11. Once returned to 1.0mL, the sample is ready for instrumental analysis.

Note: *It is very important not to concentrate to dryness as analytes will be lost.*

Note: *The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.*

11.8. Clean-up of 8270SIM-PAH Extracts

11.8.1. Use clean 20mm glass columns packed with a plug of DCM rinsed glass wool.

11.8.2. Rinse the glass wool and column with Hexane

11.8.3. Add 4cm of activated silica gel (~10g) then a 2cm layer of sodium sulfate. Tap the column while adding these reagents to prevent channeling. Elute and discard 40 mL Hexane.

11.8.4. Add the sample extract to the top of the column by quantitative transfer with 2 rinses. (The sample volume can be between 1 and 5 mL).

11.8.5. Immediately after adding the sample, elute the column with 25 mL of hexane.

11.8.6. Just before the last of the hexane hits the sodium sulfate layer, add 25 mL of hexane:methylene chloride (2:3)

11.8.7. Collect the entire elute mix in a 250mL flask.

11.8.8. Following the Kuderna Danish Concentration section in this SOP, concentrate the extract to low volume (approximately 5mL) and complete the solvent re-exchange by adding 50mL of DCM and concentration to approx 5mL.

11.8.9. Proceed to the Nitrogen Evaporation to Final Concentration section.

11.9. HLB Column Verification

Each lot of HLB cartridges must be evaluated before use, following this performance check procedure:

11.9.1. Obtain 1.0mL of a 50ug/mL mid-level solution of 8270 compounds in DCM (prepared from the GCMS calibration stock). This can be obtained from the GCMS group.

11.9.2. In a clean test tube add 1.0mL of this HLB check solution (8270 standard).

11.9.3. Add this to a pre-washed HLB cartridge.

11.9.4. Process this per the instructions in the Clean-up of 8270 Extracts section (Section 11.7). For a blank sample, repeat this step on another cartridge loaded with 2mL of clean DCM.

11.9.5. Deliver 1.0mL of the final cleaned HLB check and blank to the GCMS

instrument group for analysis.

11.9.6. Analyze the HLB check solution and blank, per method 8270. Expected recoveries are 70-120% of all analytes and surrogates with the exception of the ones listed below, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for soils. Additionally, there should be no indication of peaks interfering with target compounds.

11.9.7. Compounds with Low Recoveries Expected in HLB Checks

- Pyridine
- Aniline
- Benzoic Acid
- 4-Chloraniline
- 2,4-Dinitrophenol
- 4,6-Dinitro-2-methylphenol
- 3, 3'-Dichlorobenzidine

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures

used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/TCLP leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples and thimbles, sodium sulfate, glass wool/filter paper contaminated with methylene chloride and acetone from sonication and soxhlet extraction. Pour any excess liquid from the extracted soil samples as outlined below (Section 15.4). Dump the extracted soil and thimbles into an orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Used assorted disposable glassware and materials contaminated with methylene chloride. Dump the solid waste into a yellow contaminated lab trash bucket. When the

bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.4. Mixed flammable solvent and methylene chloride waste generated during soil extraction, glassware and sodium sulfate cleaning.. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III

Method 3500B, Organic Extraction and Sample Preparation, Revision 2, December 1996

Method 3510C, Separatory Funnel Liquid Liquid Extraction, Revision 3, December 1996

Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996

Method 3540C, Soxhlet Extraction, Revision 3, December 1996

Method 3550B, Ultrasonic Extraction, Revision 2, December 1996

Method 3580A, Waste Dilution, Revision 1, July 1992

Method 3600C, Cleanup, Revision 3, December 1996

Method 3511, Organic Compounds in Water by Microextraction, Revision 0, November 2002

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV

Method 3500C, Organic Extraction and Sample Preparation, Revision 3, February 2007

Method 3546, Microwave Extraction, Revision 0, February 2007

Method 3550C, Ultrasonic Extraction, Revision 3, February 2007

16.3. EPA Memorandum (Dated August 5, 2010) Regarding “Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods”

17. METHOD MODIFICATIONS

17.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.2. Aqueous sample volumes may be determined by weight.

17.3. Spiking for Method 3550C is performed after mixing with drying agents, as per the EPA surrogate spiking memo (Section 16.3, above).

18. ATTACHMENTS

18.1. Table 1 – 8270 List 1 Standard 1

18.2. Table 2 – Additional Stock Solutions

18.3. Table 3 – PAH-SIM Stock Standard

18.4. Table 4 – Standard Reagent Mixtures

18.5. Table 5 – PAH-SIM Isotope Dilution Analyte Solution

18.6. Table 6 – TestAmerica Sacramento 8270C Extraction Summary

18.7. Table 7 – Spiking table

19. REVISION HISTORY

19.1. WS-OP-0001, Revision 4.1, Effective 08/22/2014

19.1.1. Rearranged the Attachments to reflect the “Standard Standards” spiking mixtures and how they are implemented in the facility. Tables 1-6 (formerly the composition of the spike mixes) have been replaced by Tables 1-5. References in the text have been adjusted.

19.1.2. Removed Section 11.4 (Microextraction for Waters) as this process is no longer performed. Renumbered accordingly

19.1.3. Removed Section 11.6 (Method 3550C) and incorporated with Section 11.5. Renumbered accordingly.

19.1.4. Removed Section 11.7 (Soxhlet Extraction) as this process hasn’t been performed for some time. Renumbered accordingly.

19.1.5. Editorial changes.

19.2. WS-OP-0011, Revision 4.0, Effective 08/15/2013

19.2.1. Inserted Section 2.1.1, describing the two types of separatory funnel extraction.

19.2.2. Inserted Section 2.3 describing microwave extraction.

19.2.3. Added references for methods 3500C and 3550C (Update IV) as well as method 3546.

19.2.4. Removed sections regarding continuous liquid-liquid extraction.

19.2.5. Section 7.2, changed storage conditions of source standards to reflect manufacturer's recommended conditions.

19.3. WS-OP-0001, Revision 3.7, Effective 11/07/2012

19.3.1. Section 11.8: Separated PAH SIM and PAH SIM-ID in table.

19.3.2. Editorial changes.

19.4. WS-OP-0001, Revision 3.6, Effective 04/20/2012

19.4.1. Inserted Section 11.4 – Microextraction for water

19.4.2. Editorial changes

19.5. WS-OP-0001, Revision 3.5, Effective 7/25/2011

19.5.1. Inserted Section 7.2.1: Oasis HLB Extraction Cartridge: 500 mg pre-packed in 6mL cartridges with polyethylene frits (Waters part # 186000115, or equivalent).

19.5.2. Inserted Section 7.2.2: Activated Silica Gel: Activate for at least 1 hour at 190degC (+/- 5deg). Store at 130-200 deg C in a covered glass container.

19.5.3. Inserted Section 11.7.3, Nitrogen Evaporation to Volume for Clean-up.

19.5.4. Inserted Section 11.8, Clean-up of 8270 extracts

19.5.5. Inserted Section 11.9, Clean-up of 8270SIM-PAH Extracts

19.5.6. Inserted Appendix 1.

19.5.7. Editorial revisions.

Table 1			
Stock Standard - 8270 List 1 Standard 1			
Analyte Name	**Concentration µg/mL**	**Analyte Name**	**Concentration µg/mL**
1,1'-Biphenyl	1000	1,2,4,5-Tetrachlorobenzene	1000
1,2,4-Trichlorobenzene	1000	1,2-Dichlorobenzene	1000
1,3-Dichlorobenzene	1000	1,3-Dinitrobenzene	1000
1,4-Dichlorobenzene	1000	1,4-Dioxane	1000
1-Methylnaphthalene	1000	2,2'-oxybis[1-chloropropane]	1000
2,3,4,6-Tetrachlorophenol	1000	2,4,5-Trichlorophenol	1000
2,4,6-Trichlorophenol	1000	2,4-Dichlorophenol	1000
2,4-Dimethylphenol	1000	2,4-Dinitrophenol	2000
2,4-Dinitrotoluene	1000	2,6-Dinitrotoluene	1000
2-Chloronapthalene	1000	2-Chlorophenol	1000
2-Methylnapthalene	1000	2-Methylphenol	1000
2-Nitroanaline	1000	2-Nitrophenol	1000
3 & 4 Methylphenol	1000	3-Nitroaniline	1000
4,6-Dinitro-2-methylphenol	2000	4-Bromophenyl phenyl ether	1000
4-Chloro-3-methylphenol	1000	4-Chloroanaline	1000
4-Clorophenyl phenyl ether	1000	4-Nitroaniline	1000
4-Nitrophenol	2000	Acenaphthene	1000
Acenaphthylene	1000	Acetophenone	1000
Aniline	1000	Anthracene	1000
Azobenzene	1000	Benzo[a]anthracene	1000
Benzo[a]pyrene	1000	Benzo[b]fluoranthene	1000
Benzo[g,h,i]perylene	1000	Benzo[k]fluoranthene	1000
Benzyl alcohol	1000	Bis(2-chloroethoxy)methane	1000
Bis(2-chloroethyl)ether	1000	Biz(2-ethylhexyl) phthalate	1000
Butyl benzyl phthalate	1000	Carbazole	1000
Chrysene	1000	Dibenz(a,h)anthracene	1000
Dibenzofuran	1000	Diethyl phthalate	1000
Dimethyl phthalate	1000	Di-n-butyl phthalate	1000
Di-n-octyl phthalate	1000	Flouranthene	1000
Fluorene	1000	Hexachlorobenzne	1000
Hexachlorobutadiene	1000	Hexachlorocyclopentadiene	1000
Hexachloroethane	1000	Indeno[1,3,3-cd]pyrene	1000
Isophorone	1000	Napthalene	1000
Nitrobenzene	1000	N-nitrosodimethylamine	1000
N-nitrosodi-n-propylamine	1000	Pentachlorophenol	2000
Phenanthrene	1000	Phenol	1000
Pyrene	1000	Pyridine	1000

Table 2		
Additional Stock Standard Mixes		
Mix Name	**Analyte Name**	**Concentration ug/mL**
8270 List 1 Standard 2	3,3'Dichlorobenzidine	2000
	Benzidine	2000
	Atrazine	2000
	Caprolactam	2000
8270 List 1 Standard 7	N-Nitrosodiphenylamine	2000
8270 List 1 Standard 3	Benzoic Acid	2000
8270 List 1 Standard 8	Benzoic Acid	2000
	Indene	2000
	Benzaldehyde	2000
8270 List 2 Standard 7	Kepone	2000
1,4-Dioxane Standard	1,4-Dioxane	2000
Dimethyl Sulfoxide Standard	Dimethyl Sulfoxide (DMSO)	2000
8270 Surrogate Stock	Nitrobenzene-d5	5000
	p-Terphenyl-d14	5000
	Phenol-d5	5000
	2-Fluorobiphenyl	5000
	2-Fluorophenol	5000
	2,4,6-Tribromophenol	5000
	Nitrobenzene-d5	5000
	p-Terphenyl-d14	5000
	Phenol-d5	5000

Table 3			
PAH-SIM Stock Standard			
Analyte Name	**Concentration ug/mL**	**Analyte Name**	**Concentration mg/mL**
Naphthalene	2000	2-Methylnaphthalene	2000
1-Methylnaphthalene	2000	Acenaphthylene	2000
Acenaphthene	2000	Anthracene	2000
Fluorene	2000	Phenanthrene	2000
Fluoranthene	2000	Pyrene	2000
Benzo(a)anthracene	2000	Chrysene	2000
Benzo(b)fluoranthene	2000	Benzo(k)fluoranthene	2000
Benzo(a)pyrene	2000	Indeno(1,2,3-cd)pyrene	2000
Dibenzo(a,h)anthracene	2000	Benzo(ghi)perylene	2000

Table 4					
Standard Reagent Mixtures					
Name	**Component**	**Volume of Solution Used**	**Final Volume**	**Solvent**	**Final Concentration (range, ug/mL)**
8270 Spike 1	8270 List 1 Standard 1	20.00mL	200mL	99% MeOH (from VOA)	100 - 200
8270 Spike 2	8270 List 1 Standard 2	10.00mL	200mL	99% MeOH (from VOA)	200
	8270 List 1 Standard 7	10.00mL			
8270 Spike 3	8270 List 1 Standard 3	10.00mL	200mL	Acetone	50-150
	8270 List 1 Standard 8	5.00mL			
8270 Spike 4	8270 List 2 Standard 7	1.25mL	50mL	Acetone	50
8270 Spike 5	Custom DMSO Standard	1.00 mL	100mL	MeOH	
1,4 Dioxane Spike	1,4 Dioxane Standard	1.00mL	100mL	MeOH	20
PAH Spike	PAH Spike Standard	0.125mL	500mL	MeOH	0.5
8270 Surrogate	8270 Surrogate Standard	10.00mL	500mL	90:10 MeOH:DCM	100
1,4 Dioxane Surrogate	8270 Surrogate Standard	1.00mL	500mL	MeOH	10
PAH Surrogate	8270 Surrogate	2.50mL	500mL	MeOH	25

Table 5
Components of PAH-SIM Internal Standard (Surrogate) Solution
(Used only for PAH-SIM-ID)

Analyte Name	Concentration (µg /mL)	Analyte Name	Concentration (µg /mL)
Naphthalene-d8	20.0	Acenaphthylene-d8	20.0
Acenaphthene-d10	20.0	Fluorene-d10	20.0
Phenanthrene-d10	20.0	Fluoranthene-d10	20.0
Pyrene-d10	20.0	Benzo(a)anthracene-d12	20.0
Chrysene-d12	20.0	Benzo(b)fluroanthene-d12	20.0
Benzo(k)fluoranthene-d12	20.0	Benzo(a)pyrene-d12	20.0
Perylene-d12	20.0	Indeno(1,2,3-cd)pyrene	20.0
Dibenzo(a,h)anthracene-d14	20.0	Benzo(g,h,i)perylene-d12	20.0

TABLE 6
TestAmerica Sacramento 8270C Extraction Summary

Method (Method Code)	Sample Amount	Surrogate	LCS	pH 1 / pH 2	Extract Solvent/Vol.	Extraction Time	Comments
8270C (Aq, 1LS)	1L	0.5mL 8270 Surrogate	1.0mL 8270 Spikes	1-2 / 11-12	3 x 60mL DCM 3 x 60mL DCM	2min shake, 10min rest	Leachates use 200mL diluted to 1L with rgt water. FV=1.0mL
8270C (Solid, SONC)	30g			NA	3 x 100mL DCM:ACE 1:1	3 min	FV=1.0mL
8270C (Aq, LVI)	250mL	0.12 mL 8270 Surrogate	0.25mL 8270 Spikes	1-2 / 11-12	3 x 30mL DCM 3 x 30mL DCM	2min shake, 10min rest	FV=1.0mL
8270C (Waste Dil'n)	1 g	0.5mL 8270 Waste-Surrogate (1000ug/mL)	See table 11	NA	10mL	NA	Don't concentrate
8270C (Solid, MAE)	15g	0.12mL 8270 source	1.0mL 8270 Spikes	NA	25mL DCM:ACE 1:1	25min	FV=1.0mL
1,4-Dioxane (Aq, 1LS)	1L	500 uL 1,4 Dioxane Surrogate	500uL 1,4 Dioxane Spike	NA	3 x 60mL DCM	2min shake, 10min rest	FV=1.0mL
PAH (Waste Dil'n)	1 g	0.25 1,4 dioxane surrogate	0.5 mL 8270 L1 Std 1 20X	NA	10mL	NA	Don't concentrate
PAH (Aq, 1LS)	1L	0.5mL PAH Surrogate	0.5mL PAH Spike Mix	NA	3 x 60mL DCM	2min shake, 10min rest	FV=1.0mL
PAH (Solid, SONC,)	10 g			NA	3 x 100mL DCM	3 min	
PAH (Aq, LVI)	250mL	0.12mL PAH Surrogate	0.12mL PAH Spike	NA	3 X 30mL	2min shake, 10min rest	FV=0.5mL
PAH (Solid, MAE)	10g	0.5mL PAH Surrogate	0.5mL PAH Spike	NA	25mL DCM	NA	FV=1.0mL
PAH-ID (Aq, 1LS)	1L	100 µL of PAH –ID I.S.. (20 µg/µl)	50 µL of PAH –ID N.S. (20 µg/µl)	NA	3 x 60mL DCM	2min shake, 10min rest	FV=0.5mL
PAH-ID (Solid, SONC)	10 g			NA	3 x 100mL DCM	3 min	

1LS = 1L Separatory Funnel; LVI = LVI/RVE Separatory Funnel; MAE = Microwave; SONC = Sonication; SOX = Soxhlet;

Table 7					
Spiking Table					
Method	**Test**	**Surrogate**		**Spike**	
		Volume	**Name**	**Volume**	**Name**
1LS	8270C	0.5mL	8270 Surrogate	1.00mL	8270 Spike 1
				1.00mL	8270 Spike 2
				1.00mL	8270 Spike 3
				1.00mL	8270 Spike 4 (spike only when needed into separate LCS)
				1.00mL	8270 Spike 5 (spike only when needed into separate LCS)
1LS	1,4-Dioxane	1.00mL	1,4-Dioxane Surrogate	1.00mL	1,4-Dioxane Spike
1LS	PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike
LVI	8270C	1.2mL	1,4-Dioxane Surrogate	0.25mL	8270 Spike 1
				0.25mL	8270 Spike 2
				0.25mL	8270 Spike 3
LVI	PAH-SIM	0.12mL	PAH Surrogate	0.12mL	PAH Spike
MAE	8270C	0.5mL	8270 Surrogate	1.00mL	8270 Spike 1
				1.00mL	8270 Spike 2
				1.00mL	8270 Spike 3
MAE	PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike
3550B/C	8270C	0.5mL	8270 Surrogate	1.00mL	8270 Spike 1
				1.00mL	8270 Spike 2
				1.00mL	8270 Spike 3
3550B/C	PAH-SIM	0.5mL	PAH Surrogate	0.5mL	PAH Spike
Waste	8270C	0.12mL	8270 Waste Surrogate	1.00mL	8270 List 1 Standard 1
				0.5mL	8270 List 1 Standard 2
				0.5mL	8270 List 1 Standard 7
				0.5mL	8270 List 1 Standard 8
Waste	PAH-SIM	0.25mL	1,4-Dioxane Surrogate	0.5mL	8270 List 1 Standard 1 20X

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Title: Extraction and Cleanup of Organic Compounds From Waters and Soils, Based on SW--846 3500 Series and 3600 Series Methods for Analysis by Methods 8081A and 8082 [Methods 3500 Series and 3600 Series]

Approvals (Signature/Date):

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Lisa Stafford — Date 4/24/2014 Interim Quality Assurance Manager	Robert Hrabak — Date 4/25/14 Laboratory Manager

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1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, TCLP leachate, soil and waste matrices for analysis by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA).

1.1. Extraction procedures for the determinative methods 8081A and 8082 are covered.

1.2. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

1.3. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

1.4. Specific handling procedures for tissue samples are found in WS-WI-0018.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction: A measured volume of sample is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.1.1. This SOP has procedures for extracting both the traditional sample volume (1000mL) as well as reduced sample volumes (250 or 125 mL). The traditional volume is referred to as a "1 L separatory funnel extraction", and the smaller volumes are referred to as "250 mL Limited Volume Initiative/Reduced Volume Extraction" (250 mL LVI/RVE) and "125 mL Limited Volume Initiative/Reduced Volume Extraction (125 mL LVI/RVE) in this SOP.

2.2. Sonication Extraction: A measured weight of sample, typically 30 g (10 g for biologics), is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted with 1:1 methylene chloride: acetone three times using an ultrasonic horn. In pre-approved situations, other solvents or solvent combinations may be used to control co-extracted matrix interferences.

2.3. Soxhlet Extraction: A 15 g sample is mixed with anhydrous sodium sulfate to form a fee flowing powder. This is extracted with refluxing 1:1 methylene chloride:acetone for 16-24 hours.

2.4. Waste Dilution: A measured weight of sample (typically 1 g), is diluted to 10 mL with hexane which is now ready for cleanup.

2.5. Microwave Extraction: A measured weight of sample, typically 15 g (10 g for biologics), is extracted with 1:1 methylene chloride: acetone.

2.6. Cleanup and Concentration: Procedures are presented for removing interferents from sample extracts, and for drying and concentrating the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3. To prevent analyte loss, extracts which are not immediately concentrated by KD must be protected from light during storage. Extracts may be either refrigerated or stored in an opaque container.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The PCB solutions used to prepare standards for this method may arrive at concentrations as high as 1,000 ppm. Exercise extreme caution when handling these high concentration standards.

5.1.2. If any PCB standards are spilled inside or outside of a fume hood at concentrations equal to or greater than 5 PPM, all materials in contact must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS Coordinator for specific disposal instructions before cleaning up the spill.

5.1.3. If any PCB standards are spilled outside the fume hood at concentrations greater than 200 ppm, the Emergency Response Team must be activated for special clean-up procedures, and possibly additional testing of the spill location and additional regulatory notifications.

5.1.4. All glassware and solid lab trash in contact with any PCB's at concentrations equal to or greater than 5 PPM must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS Coordinator for specific disposal instructions before disposing of this waste.

5.1.5. Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Microwaves used for these processes must be equipped with an automated vapor sensor and shutoff system that automatically shuts down the digestion/extraction process when vapor pressure reaches pre-specific levels. Users must follow procedures in the microwave operator's manual to ensure that the vapor sensors are functional and working properly prior to starting each extraction/digestion batch.

5.1.6. Hearing protection is required when sonicating samples.

5.1.7. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.8. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.9. Laboratory procedures such as repetitive use of pipettes, repetitive transferring

of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.10. Sulfuric acid cleanup must not be performed on any matrix that may have water or acetone present as a violent reaction between the acid and water or acetone may result in acid exploding out of the vessel.

5.1.11. Mercury is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and cleanup techniques before handling this material. Spilled mercury requires that special cleanup tools and procedures be used. .

5.1.12. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar® or similar cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware. This includes when seating Teflon stoppers into glass separatory funnels.

5.1.13. The use of vacuum systems during Florisil cartridge cleanup presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.14. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.1.15. Ensure that the vacuum exhaust hose used during the Florisil cartridge cleanup is securely anchored inside of a fume hood so that solvent vapors are not pumped into the working environment.

5.1.16. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the process may be performed behind a closed fume hood sash. After each of the three extraction cycles, ensure that the

stoppers are removed from the top of the separatory funnels.

5.1.17. When Soxhlet extractions are performed overnight or unattended, special precautions must be taken. Open chiller values to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.1.18. The kiln used to clean glassware creates an extreme risk of severe burns. Allow sufficient time for the kiln to cool before opening the doors. Ensure that appropriate heat protective gloves are worn when working with freshly kilned glassware. Other surfaces in the kiln may remain hot, so exercise caution when reaching into the kiln.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table**. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Mercury	Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. **May affect the central nervous system.** Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m3	This material will cause burns if it comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydradator	1 mg/m3	This material will cause burns if it comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Glassware is kilned at 400°C for at least 2 hours. Extremely dirty glassware can be cleaned with soap and water prior to using the kiln. Refer to SOP WS-OP-0011 for details of glassware cleaning.

Heat resistant gloves must be used when handling kilned glassware.

6.2. Equipment and supplies for extraction procedures

6.2.1. Separatory funnel, 2L (Teflon or glass)

6.2.2. Separatory funnel, 250mL (Teflon or glass)

6.2.3. Separatory funnel, 500mL (Teflon or glass)

6.2.4. Separatory funnel rotator with timer

6.2.5. Balance: >1400 g capacity, accurate to ±0.1 g.

6.2.6. pH indicator paper, wide-range: covers extraction pH.

6.2.7. Graduated cylinder: 1 liter, other sizes may be used.

6.2.8. Erlenmeyer flask, beaker, or french square jar: 125 & 300 mL (other sizes optional).

6.2.9. Solvent dispenser pump or 100 mL graduated cylinder.

6.2.10. Round or flat bottom flask: 250, 500 mL or 1 L.

6.2.11. Boiling chips: contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).

6.2.12. Cooling condensers.

6.2.13. Heating mantle: rheostat controlled, with timer.

6.2.14. Beakers: 250 & 400 mL, graduated

6.2.15. Soxhlet extractor.

6.2.16. Balance: >100g capacity, accurate to ±0.01g

6.2.17. Sonicator (at least 300 watts), with sonicator horn, 3/4 inch. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2.18. Kuderna-Danish (KD) apparatus: 500mL, with 10mL concentrator tube, and 3-ball macro Snyder column.

6.2.19. Locally assembled solvent vapor condensing/recovery system for use with 6.2.17 above.

6.2.20. Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.

6.2.21. Nitrogen blowdown apparatus.

6.2.22. Culture tubes: 16 mL, 16 mm x 125 mm

6.2.23. Syringe: 1 mL.

6.2.24. Glass wool.

To ensure glass wool is free of contaminants, it is necessary to soxhlet rinse glass wool in methylene chloride for 18 hours.

6.2.25. Glass funnel: 75 X 75 mm.

6.2.26. Disposable pipettes.

6.2.27. Aluminum foil.

6.2.28. Paper towels.

6.2.29. Pressure rotor and pressure reactor

6.2.30. Extraction vessel and extraction vessel cover

6.2.31. Glass inserts

6.2.32. Microwave – Milestone Ethos EX

6.3. Equipment and Supplies for Cleanup Procedures

6.3.1. Vacuum system for eluting multiple cleanup cartridges. Vac elute manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.

6.3.2. Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.

6.3.3. Vacuum pressure gauge.

6.3.4. Rack for holding collection vessels in the manifold.

6.3.5. Mechanical shaker or mixer: vortex genie or equivalent.

6.3.6. Disposable pipettes

6.3.7. Culture tubes: 8 mL, 13 mm x 100 mm

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

7.1.1. All reagents must be ACS reagent grade or better unless otherwise specified.

7.1.2. Sodium hydroxide (NaOH) solution: 10N reagent, grade.

7.1.3. Sulfuric acid (H_2SO_4), concentrated; reagent grade.

7.1.4. Sulfuric acid (1:1): carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.

WARNING: Always add concentrated acids or bases to water, never add water to the concentrated acid or base solution. Due to the reaction between acid and water, add the acid slowly to water to prevent breaking of the mixing vessel

7.1.5. Organic free reagent water (deionized water). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks, in accordance with SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water.

7.1.6. Sodium sulfate (Na_2SO_4), granular, anhydrous, reagent grade: purify by heating at 400°C for a minimum of two hours.

7.1.7. Extraction/exchange solvents: methylene chloride, hexane, acetone, pesticide quality or equivalent

7.1.8. Acetone: used for cleaning

7.1.9. Nitrogen, reagent grade

7.2. Reagents for Cleanup Procedures

7.2.1. Florisil: 500 mg or 1 g cartridges with stainless steel or PTFE frits (JT Baker cat # 7420-07 or equivalent)

7.2.2. Styrene Divinylbenzene polymer sorbent: 500 mg pre-packed in 6mL polypropylene cartridges with polyethylene frits (Phenomenex, cat # 8B-S014-HCH, or equivalent).

7.2.3. Graphitized Carbon sorbant cleanup: 500 mg pre-packed in 6mL polypropylene cartridges with polyethylene frits (Restek "CarboPrep-90", cat # 26092 or equivalent).

7.2.4. Mercury: triple distilled.

7.2.5. Sulfuric acid, concentrated.

7.3. Standards

7.3.1. Source Standards

7.3.1.1. Source standards are purchased as certified solutions or prepared from neats. Semivolatile source standards are stored at room temperature. All stock standards must be protected from light.

7.3.1.2. Source standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Standards must be allowed to come to room temperature before use.

7.3.1.3. Expired standards must be rotated out of the lab to the Hazardous Waste storage area.

7.3.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 3. Surrogate spiking standards are prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the Matrix Spike (MS), Matrix Spike (MSD), and the Laboratory Control Sample (LCS). Prepare MS/LCS spiking standards at the concentrations listed in Table 3. Spiking standards are purchased or prepared as dilutions of the source standards. Spiking solutions must be refrigerated and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Endrin aldehyde and BHC standards are given a 2 week expiration date from the time of preparation. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4 ± 2°C in glass containers with Teflon®-lined caps

8.3. Holding Times

8.3.1. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2. For TCLP leachates, extraction is initiated within seven days from when the leaching procedure is completed.

8.3.3. Analysis of the extracts must be completed within forty days of extraction.

9. QUALITY CONTROL

Quality Control Batch: The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a MB, an LCS and a MS/MSD. (In some cases, at client request, it may be appropriate to process a

matrix spike and sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy WS-PQA-003 for further definition of the batch.

9.1. Definition of Matrix
The possible matrix types are aqueous, solid, waste, TCLP leachate, and biologics. Refer to WS-WI-0018 for tissue sample handling.

9.2. Insufficient Sample
If insufficient sample is available to process a MS/MSD, a second LCS may be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Use of a LCS pair in place of a MS/MSD must be documented using the non-conformance memo (NCM) program in TALS.

9.3. Sample Count
Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.4. Method blanks for biologic samples (fish, plants, worms, etc.) normally use 10 g of sodium sulfite spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5. Method Blank
A MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1. Aqueous MB use 1000 mL for 1L separatory funnel extraction, 125 mL for 125 mL LVI/RVE, and 250 mL for 250 mL LVI/RVE of organic free reagent water (deionized water) spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2. Solid method blanks use 30 g of kilned sodium sulfate for 3550B and 15 g Ottawa sand for 3546 spiked with the surrogates. The MB goes through the entire analytical procedure, including any cleanup steps.

9.5.3. TCLP and STLC MB use 200 mL of leachate fluid spiked with the surrogates. Dilute to 1000mL with organic free reagent water (deionized water). The MB goes through the entire analytical procedure, including any cleanup steps.

9.5.4. MB for waste dilution procedures consist of 1 g of vacuum oil spiked with the surrogates. The MB goes through the entire analytical procedure, including

any cleanup steps.

9.5.5. MB for tissue consists of 200 uL fish oil (Sigma F8020-500mL) mixed with either 10 g sodium sulfate (3550B/C) or 10 g Ottawa sand (3546).

9.6. Laboratory Control Sample (LCS)

LCS is a well-characterized, laboratory generated sample used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1. The LCS is made up in the same way as the MB (See Sections 9.5.1 - 9.5.5), but is spiked with the LCS standard and the surrogate.

9.7. Surrogates

9.7.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2. Each applicable sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS is an environmental sample to which known concentrations of target analytes have been added. A MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. The MS and MSD are spiked with the LCS standard and the surrogate.

9.9. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in section 13 must be acceptable before analysis of samples may begin.

9.10. Quality Assurance Summaries Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.11. West Sacramento QC Program Further details of QC and corrective action guidelines are presented in the Sacramento QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. On the day of use, measure 2.0 mL of solvent into a 16mL culture tube using a 1.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Seal the culture tube – this is the 'check' or 'reference' vial for cleanup with Florisil. Using the same procedure, create a 10.00mL 'check' vial using a 5.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Record the container lot, solvent, gastight syringe or serological pipet ID, date, and initials of preparer for all check vials in a controlled logbook. The culture tubes containing the sample extracts are then compared against the "check" vial to ensure that the final volume is consistently within the criteria of 10 ± 0.3 or 2 ± 0.06 mL as required.

10.2. On a daily basis, calibrate any auto-pipettors to be used in accordance with SOP WS-QA-0004.

10.3. On the day of use, calibrate any balances to be used in accordance with SOP WS-QA-0041.

10.4. Prior to use, ensure any containers used for quantititative steps (e.g final volume and Florisil) are verified as described in SOP WS-QA-0004.

10.5. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed, or annually, whichever is sooner.

10.5.1. Newer models of sonicators may be self-tuning. Read the manufacturer's directions prior to operating this equipment.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (Section 11.2), sonication extraction (Section 11.3), waste dilution (Section 11.4), extract concentration (Section 11.5), and extract cleanup (Section 11.6) are presented in this section.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Method 3510C, Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and LCS spiking standards from refrigerator and allow standards to warm to room temperature.

11.2.1.1. If the sample bottle contains a layer of sediment greater than approximately ½" in depth, contact the department manager and the project manager to determine how to proceed with the sample extraction. Options may include the following:

a) Decanting and extracting only the liquid fraction in a separatory funnel before adding the surrogate and/or spike solutions.
b) Adding the surrogate and/or spike solutions to the sample with the sediment still in the original sample container.
c) Processing the aqueous portion as described in a) along with creating a new sample id for the soil matrix. The sample bottle would then be rinsed with 60 mL of DCM, spiked with an appropriate volume of surrogate solution and shaken for 3 minutes. The addition of 60 mL of DCM will be repeated 2 more times and all solvents combined to create a second extract for the sample that is to be added to the extraction batch of water samples

NOTE: In the case of option "b", after adding the appropriate surrogates/spikes, the sample would be gently swirled for mixing purposes and then gently poured into the separatory funnel, thus minimizing the amount of sediment transferred to the extraction v vessel during the process. The original sample container would then be rinsed with 60 mL of DCM. This DCM rinseate would then be poured into the extraction vessel.

11.2.2. Measure the initial sample pH with wide-range pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against the leachate log and note on the benchsheet if there is any discrepancy.

11.2.3. The normal sample volume is 1 liter for 1L separatory funnel extraction, 125 mL for 125 mL LVI/RVE, and 250 mL for 250 mL LVI/RVE. Other sample volumes may be used to obtain specific reporting limits, dirty samples, and reduced sample volumes, diluted to 1 liter, 125mL, or 250 mL with organic free reagent water (deionized water).

11.2.4. Obtain sample weight(s) to ± 0.1 g. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. This may be done in two ways depending if a dilution is necessary.

11.2.4.1. For samples not requiring a dilution weigh the gross container on a tared balance and after extraction has been initiated weigh the tared container on a tared balance and use the difference as the sample weight.

11.2.4.2. For samples requiring a dilution, subsample the desired aliquot amount in accordance with SOP-QA-0018 into a tared container. Dilute to 1L nominal for 1L separatory funnel extraction and 250mL nominal for 250 mL LVI/RVE and 125 mL nominal for 125 mL LVI/RVE with organic free reagent water (deionized water). This must be documented in an NCM, as there may be a low bias to the analysis when the entire container is not use, and the container is therefore not rinsed and the rinsate combined with the extraction solvent.

11.2.4.3. For TCLP samples, use 200mL of leachate and dilute to 1L in organic free reagent water.

Note: Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.

11.2.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP. See Tables 3 and 4 for spiking volumes and concentrations.

11.2.6. For a TCLP batch prepare a MB as specified in section 9 of this SOP. In addition, prepare a TCLP method blank by measuring 200mLs of buffer solution used in the leaching procedure and dilute to 1L nominal using the method described in 11.2.4. No leachate LCS is required by this method.

11.2.7. Adjust sample pH to between 5 and 9. Use the minimum amount of 1:1 H_2SO_4 or 10N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper.

11.2.8. Record adjusted pH, spiking volumes standard numbers, and reagent lots on the benchsheet.

11.2.9. After samples have warmed to room temperature, add the surrogate standard to all samples and the LCS solution to the necessary samples as described in section 9 of this SOP. See Tables 3 and 4 for spiking volumes and concentrations. Return spiking solutions to the refrigerator as soon as possible after spiking.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer part of the sample to the separatory funnel and then add the spike to the bottle.

11.2.10. Mix well, or if the sample contains sediment, decant before transfer. Transfer the sample to the separatory funnel. Rinse the sample bottle with 60 mL methylene chloride for 1L separatory funnel extraction. 30 mL for 250 mL LVI/RVE and 15 mL for 125 mL LVI/RVE. Transfer the rinsate into the 2L glass or Teflon separatory funnel for 1L separatory funnel extraction, 500 mL glass or Teflon separatory funnel for 250 mL LVI/RVE, or 250 mL glass or Teflon separatory funnel for 125 mL LVI/RVE.

11.2.11. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. As described in 5.1.11, Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle.

***WARNING:* Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.**

11.2.12. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (at least 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.2.13. One method for reducing the effects of emulsions is to swirl the funnel after allowing it to rest for the required amount of time, wait approximately a minute, and then drain the extract without collecting the emulsion. After the third (final) extraction and rest period, add a squirt of DCM to identify where the layer of DCM and water is separated and collect only the DCM. Alternatively, on the final pour collect the emulsion and use a second filtration step at KD with glass wool and pipet off excess water.

***Note:** 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 40 mL from the first shake and still be acceptable for 1L separatory funnel extraction. Subsequent shakes should recover at least 50 mL of solvent for 1L separatory funnel extraction. Similarly, it is anticipated that only 20 of the 30 mL used (or 10 of 15 mL used) may be recovered on the first shake when extracting smaller water volumes.

11.2.14. Fill a funnel with anhydrous sodium sulfate (10-15g for LVI/RVE, nearly a

full funnel for 1L separatory funnel extraction). The funnel can be plugged with glass wool to hold the sodium sulfate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just ***before*** the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate prior to the concentration step. Ensure to minimize the amount of water collected.

11.2.15. Repeat the extraction process two more times using fresh portions of solvent (60 mL for 1L separatory funnel extraction, 30 mL for 250 mL LVI/RVE, and 15 mL for LVI/RVE) combining the three solvent extracts in the collection container.

11.2.16. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.17. Dispose of solvent and water remaining in the extractor into the LLE waste drum. Waste methylene chloride goes to the waste solvent drum for disposal.

11.2.18. Cap the collection container and refrigerate the container if the extract is not concentrated immediately. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.3. Method 3546, Solid Extraction by Microwave

11.3.1. Remove surrogate and matrix spiking solutions from refrigerator and allow standards to warm to room temperature.

11.3.2. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.3.3. Weigh 15 g of sample (10 g for biologics) ± 1.0 g into an extraction vessel. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

11.3.3.1. For visually dirty samples, it may be necessary to weigh the sample into a glass insert to be placed into an extraction vessel. Contact your supervisor to determine if this is the case.

11.3.4. Mix weighed sample with a spatula. If clay clumps or other sample

aggregation is evident, add Ottawa sand (not to exceed 5 g) as needed to facilitation disaggregation and mix as necessary.

11.3.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.3.6. Add the surrogate spiking solution to each sample, MB, LCS, and MS/MSD. Add the appropriate matrix spiking solution to each MS/MSD and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.3.7. Immediately add 25 mL of 1:1 hexane:acetone to extraction vessels.

Note: Steps 11.3.6 - 11.3.7 should be performed rapidly to avoid loss of the more volatile extractables.

11.3.8. Add flared extraction vessel covers to each extraction vessel containing sample and QC aliquots.

11.3.8.1. It is important that each cover fits snugly to ensure a proper seal. The cover should not slide easily or loosely inside the extraction vessel, but should require some finger pressure to insert firmly. A cover flaring tool should be used.

11.3.8.2. For the visually wettest sample, add the thermowell liner into the extraction vessel cover to create the representative sample that the ATC temperature sensor can be inserted into.

11.3.9. Place each extraction vessel into a pressure reactor. Screw on the pressure cap/safety lid. The pressure cap should be hand tightened until the sealing valve is flush with the top of the cap.

11.3.9.1. For the representative sample created in 11.3.8.2, add the protection foil and appropriate safety lid.

11.3.10. Place all the extraction vessels into the rotor so that the pressure-release valves are facing outside of the rotor on the outside ring and inside toward the center on the inside ring.

WARNING: Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Follow procedures in the operator's manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

11.3.10.1. Place the rotor in the microwave oven and insert the ATC temperature sensor into the representative sample (11.3.8.2 and 11.3.9.1).

11.3.11. Close the microwave oven and start the appropriate extraction profile.

11.3.11.1. A 10 minute ramp from 25C to 100 C, hold for 25 minutes at 100C, followed by a 15 minute cool down to 25C

11.3.12. After the extraction period and cool down, pressure reactors should be at ambient temperature prior to removal from the rotor and opening.

11.3.13. Proceed to Section 11.8 for concentration and Section 11.9 for cleanup.

11.3.13.1. Discard any glass inserts. To clean extraction vessels and pressure cap/safety lid, use soap and water followed by a solvent rinse for. Use only solvent to rinse the probe. Allow to air dry prior to use.

11.4. Method 3550B, Solid Extraction by Sonication

11.4.1. Sonicator Tuning

11.4.1.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed, or annually, whichever is sooner.

11.4.1.2. Newer models of sonicators may be self-tuning. Read the manufacturer's directions prior to operating this equipment.

11.4.2. Remove surrogate and matrix spiking solutions from refrigerator and allow solutions to warm to room temperature.

11.4.3. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.4. Weigh 30 g of sample (10 g for biologics) ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate (10 g for biologics) for the method blank and LCS.

11.4.5. Mix weighed sample with a spatula or blending rotor, adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.6. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.4.7. Add the surrogate spiking solution to each sample, MB, LCS, and MS/MSD. Add the appropriate matrix spiking solution to each MS/MSD and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.4.8. Immediately add a minimum of 100 mL of 1:1 DCM/acetone to the beaker. In pre-approved situations, other solvents (such as Hexane or DCM only) may be used to control co-extracted matrix interference).

Note: Steps 11.4.7 - 11.4.8 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.9. Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pour beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horns with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

11.4.10. Place the bottom surface of the sonicator disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.11. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

WARNING: Hearing protection is required when sonicating samples.

Note: Do ***not*** use ***Microtip*** probe.

11.4.12. Decant extracts into a clean beaker.

11.4.13. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication.

11.4.14. Cap the collection container. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.5. Method 3550C, Solid Extraction by Sonication

Method 3550C differs from 3550B in the order in which samples are dried and spiked. Be certain that the method performed reflects the method requested in the LIMS (TALS).

11.5.1. Sonicator Tuning

11.5.1.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed, or annually, whichever is sooner.

11.5.1.2. Newer models of sonicators may be self-tuning. Read the manufacturer's directions prior to operating this equipment.

11.5.1.3. ***Note:*** Do ***not*** use ***Microtip*** probe.

11.5.2. Remove surrogate and matrix spiking solutions from refrigerator and allow solutions to warm to room temperature.

11.5.3. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.5.4. Weigh 30 g of sample (10 g for biologics) ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate (10 g for biologics) for the method blank and LCS.

11.5.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.5.6. Add the surrogate spiking solution to each sample, MB, LCS, and MS/MSD. Add the appropriate matrix spiking solution to each MS/MSD and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.5.7. Mix weighed sample with a spatula or blending rotor, adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.5.8. Immediately add a minimum of 100 mL of 1:1 DCM/acetone to the beaker. In pre-approved situations, other solvents (such as Hexane or DCM only) may be used to control co-extracted matrix interference).

Note: Steps 11.5.6 - 11.5.8 should be performed rapidly to avoid loss of the more volatile extractables.

11.5.9. Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pour

beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horns with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

11.5.10. Place the bottom surface of the sonicator disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.5.11. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

WARNING: Hearing protection is required when sonicating samples.

11.5.12. Decant extracts into a clean beaker.

11.5.13. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication.

11.5.14. Cap the collection container. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.6. Method 3540C, Extraction of Solids with Soxhlet Apparatus

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensers are cold before you turn the heating element on. Check all of the condensers above 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this extraction cycle is to be left unattended (e.g., overnight), the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.6.1. Decant and discard any waste layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials, consult with the client. Document on the benchsheet if a water layer was discarded.

11.6.2. Remove surrogate and matrix spiking solutions from refrigerator and allow solutions to warm to room temperature.

11.6.3. Weigh 15 g of sample ± 1.0 g into a beaker or glass jar, recording the weight to the nearest 0.1 g on the benchsheet. Use 15 g of sodium sulfate for the method blank and LCS. Add 15 g of anhydrous sodium sulfate to the samples

and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction period.

11.6.3.1. Sample weights less than 15 g but over 5 g may be used if the appropriate reporting limits can be met.

11.6.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.6.5. Add the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Dilution (MS/MSD) and LCS. Refer to Tables 3 – 6 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.6.6. Place approximately 300 mL of 1:1 methylene chloride/acetone into a 500 mL round bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16–24 hours at 4–6 cycles per hour. The flask should be heated by setting the controller to 3.5 on the dial. Check the system for leaks at the ground glass joints after it has warmed up. In pre-approved situations, other solvents (such as Hexane or DCM) may be used to control co-extracted matrix interference).

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensers are cold before you turn the heating element on. Check all of the condensers about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If the cycle is to be left unattended (e.g. overnight) the first analyst to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.6.7. Allow the extract to cool after the extraction is complete. Collect all solvent in the round bottom flask. Disassemble by gently twisting the soxhlet from the flask. Do not force the glassware apart.

WARNING: Use of cut-resistant gloves (Kevlar®) or similar material) is required when assembling or disassembling glassware.

11.6.8. Collect the dried extract in a KD or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and

add it to the funnel to complete the quantitative transfer.

11.6.9. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.7. Method 3580A, Waste Dilution

11.7.1. This method is used for materials that are soluble in an organic solvent.

11.7.2. Remove surrogate and matrix spiking solutions from refrigerator and allow the standards to warm to room temperature.

11.7.3. Tare the vial, then transfer approximately 1 g of sample to the vial. Record the weight to the nearest 0.01 g.

11.7.4. Add the surrogate solution (Table 3).

11.7.5. Dilute to 10 mL with hexane.

11.7.6. Prepare the necessary QC (MB/LCS/LCSD/TOX) samples by adding 1 g of clean oil matrix (vacuum oil or equivalent) to clean 16 mL culture tubes. Add surrogate and matrix spike solution (Table 3) to the appropriate QC samples. Dilute the contents of each tube to 10 mL with hexane.

11.8. Concentration: According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 1 for the appropriate final volumes and concentrations.

11.8.1. Kuderna-Danish (KD) Method

11.8.1.1. For aqueous samples, loosely plug the stem of a 75mm x 75mm glass funnel with glass wool and rinse well with DCM. For solid samples place a folded piece of Whatman #1 filter paper into the glass funnel.

11.8.1.2. For LVI/RVE samples, add approximately 10-15 g of anhydrous sodium sulfate to the funnel cup. For 1L separatory funnel extraction, add anhydrous sodium to nearly fill up the funnel cup. For solid samples, add approximately 5-10 g of anhydrous sodium sulfate to the funnel cup.

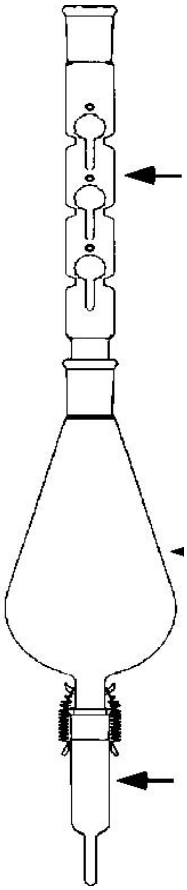
11.8.1.3. Pour the sample through the prepared glass funnel into the KD flask. Add one or two clean boiling chips to the KD flask.

11.8.1.4. Rinse the top of the funnel as well as the sample container with DCM.

11.8.1.5. Prepare the three ball Snyder columns by rinsing them with DCM to ensure that the balls are not stuck and that the column will work properly.

WARNING: Use of cut-resistant gloves (Kevlar® or similar material) is required when assembling or disassembling glassware.

Figure 2, KD Setup (See Section 11.6.1 for description)



11.8.1.6. Place the KD apparatus on a water bath (85-90°C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood. Attach the solvent vapor recovery system to the top of the Snyder column.

WARNING: Do not concentrate to dryness as this may cause flammable vapor in the KD flask to superheat and explode.

11.8.1.7. When the apparent volume of the extract reaches 10-15 mL, remove the KD apparatus from the water bath and allow it to drain and cool

11.8.1.8. For samples to be loaded o the Gel Permeation Chromatograph (GPC), adjust the final DCM volume to 10 mL and split this into two, 5mL portions. Archive one of the 5 mL fractions and GPC the other.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.8.1.9. To solvent exchange to hexane, momentarily remove the Snyder column, add 50 mL of hexane, a new boiling chip, and reattach the Snyder column. Alternatively, pour the exchange solvent into the top of the Snyder column. For LVI/RVE, add only 25 mL of hexane. No solvent exchange is necessary for MAE.

11.8.1.10. Put the KD assembly back on the water bath and concentrate the extract to ~5mLs, as described in the previous sections, raising the temperature of the water bath, if necessary, to maintain proper distillation

11.8.1.11. Remove the KD apparatus from the water bath and allow it to cool and drain. If the level of the extract is above the level of the concentrator tube joint, add a new boiling chip and continue to distill the solvent as necessary. If redistilling, remove the KD apparatus from the water bath and allow it to cool and drain before proceeding to the next step.

11.8.1.12. Remove the Snyder column and rinse the inside of the top joint of the K-D flask with a minimum amount of DCM, rotating the flask as necessary to ensure the rinsate covers the sides of the KD flask.

Note: When performing KD on the GPC portion of the extract, bring to a final volume of 5 mL (as the 10 mL extract was split in the previous Section).

11.8.1.13. If further concentration is indicated in Table 1, either the micro Snyder column technique or nitrogen evaporation (Section 11.6.2) technique is used to adjust the extract to the final volume required.

11.8.2. Nitrogen Evaporation (N-evap) to Final Concentration

11.8.2.1. Quantitatively transfer the extract into a calibrated container. Alternatively, place the concentrator tube in a warm water bath that is at least 5° C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract. Hexane boils at 69°C.

11.8.2.2. During the course of the evaporation rinse the sides of the evaporation tube or container twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 10 mL.

11.8.2.3. When the volume is slightly below 10 mL, remove the container/tube from the n-evap bath. Quantitatively transfer the extract into a calibrated container if the extract is still in the concentrator tube. Compare the volume to that of the calibrated reference, and carefully add solvent to make the final volume of 10 mL. Record the lot of the container that extracts are brought to final volume in.

Note: It is very important not to concentrate to dryness as analytes will be lost.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.8.3. Proceed to the cleanup steps.

11.9. Cleanup Techniques

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/or loss of detector sensitivity.

11.9.1. *Gel Permeation Chromatography* (SOP WS-OP-0012, Method 3640A) is a generally applicable technique which can be used to prepare extracts for semivolatiles (8270), and pesticides/PCB analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.

Note: Gel Permeation Chromatography is generally the first cleanup step.

11.9.2. *Florisil column cleanup* (Section 11.10, Method 3620B) is particularly useful for cleanup of pesticides for analysis by Methods 8081A/8081B and PCBs by Method 8082/8082A and should normally be applied to these samples unless the matrix is clean. It separates compounds with a different polarity from the target analytes.

11.9.3. *Styrene Divinylbenzene polymer (SDB-L) sorbant cleanup* (Section 11.11). The SDB-L Styrene-Divinylbenzene polymer SPE tubes (Phenomenex) or DVB SPE tubes (Restek) may be helpful for cleanup of pesticides for analysis by Methods 8081A/8081B and PCBs by Method 8082/8082A. This cleanup should be applied routinely to all extracted soil samples. Use on other matrices is also appropriate if co-extracted contamination is problematic at instrument analysis. This synthetic polymer is used to remove a variety of

unwanted matrix-related compounds.

11.9.4. *Graphitized Carbon sorbent cleanup* (Section 11.12). The CarboPrep-90 (Restek) or EnviCarb (Supelco) pre-packed cartridges may be helpful for cleanup of pesticides for analysis by Method 8081A and PCBs by Method 8082. This clean-up is not applied routinely as there is approximately 50% recovery of TCX surrogate. Its use should be pre-approved by the department manager. This clean-up removes oils and humic substances.

11.9.5. *Sulfuric acid cleanup* (Section 11.13, Method 3665A) is applied to samples requiring analysis for 8082/8082A Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by the sulfuric acid. In pre-approved situations, this acid cleanup can be used for 8081A/8081B samples, however, there will be significant loss (or increase) of some compounds, namely: Aldrin, Dieldrin, Endrin, Endosulfan I, Endosulfan II, Endrin Aldehyde, Methoxychlor, Endrin Ketone, and Endosulfan Sulfate

WARNING: Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

11.9.6. *Sulfur cleanup* (Section 11.14, Method 3660A) is generally applied to samples for analysis by methods 8081A/8081B and 8082/8082A, since the Electron Capture Detector responds strongly to sulfur. It is performed as the last cleanup.

11.10. Method 3620B, Florisil Cartridge Cleanup

Note: Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assembly, Zymark Benchmate, or equivalent.

11.10.1. Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.10.1.1. Place a valve liner (Supelco, P/N# 57059, disposable flow control valve liner, or equivalent) into each port of the vacuum manifold.

WARNING: Use of vacuum systems creates a significant risk of implosion. Thoroughly inspect all glassware and do not use any that has been chipped, rubbed, cracked, or marred in any fashion.

WARNING: Ensure that the exhaust line from the vacuum pump is secured well inside of a fume hood so that it cannot fall out of the hood.

11.10.2. Place one Florisil cartridge into the vacuum manifold for each sample extract.

11.10.3. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of hexane/acetone (1:1). Rinse with 5mL of hexane. Adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min. Do not allow the cartridges to go dry.

11.10.4. Just before the cartridges go dry, release the vacuum to the manifold and remove the manifold top.

11.10.5. Place a rack of clean labeled 16 mL test tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.10.6. After the clean tubes are in place, vacuum to the manifold is restored, add 2.0 mL (1L separatory funnel extraction and 3550B/3550C) or 1.5 mL (LVI/RVE and MAE) of the extract to the appropriate Florisil cartridge. Archive the remaining extract.

11.10.7. The pesticides/Aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 16 mL test tube or held in the rack inside the vacuum manifold. Allow the solvent to drip through the sample without vacuum until drip stops. Then apply vacuum to extract remaining eluent.

11.10.8. Use nitrogen evaporation (Section 11.6.2) to concentrate the extract to the same amount that was used in 11.8.6. Afterward the sample extract can proceed to the subsequent clean-up steps in the following sections.

11.10.8.1. Florisil lot verification procedure – see Section 11.15.

11.11. Divinylbenzene Styrene Polymer Cartridge Cleanup

11.11.1. This clean-up should be conducted simultaneously with Florisil cleanup. Follow the same elution procedure as the Florisil cleanup from the previous section. The SDB (dimethylbenzene styrene) cartridge should be stacked on top of the Florisil cartridge referenced in Section 11.10.3.

11.11.2. Each lot should be verified for recovery of target analytes using the same procedure as that for Florisil ((Section 11.15). The evaluation of 2,4,5-TCP recovery is omitted for this clean up material.

11.12. Graphitized Carbon Cartridge Cleanup

11.12.1. This clean-up should be conducted after Florisil (if approved). Follow the same elution procedure as the Florisil cleanup from the previous section.

11.12.2. Each lot should be verified for recovery of target analytes using the same

procedure as that for Florisil (Section 11.15). The evaluation of 2,4,5-TCP recovery is omitted for this clean up material.

11.13. Method 3665A, Sulfuric Acid Cleanup

11.13.1. Add approximately 2 mL of concentrated sulfuric acid to 2.0 mL (1L separatory funnel extraction and 3550B/3550C) or 1.5 mL (LVI/RVE and MAE) sample extract in a Teflon capped vial.

WARNING: There must be no water present in the extract or the reaction may shatter the sample container.

11.13.2. Shake or vortex for about thirty seconds and allow it to settle. Centrifuge if necessary.

11.13.3. Remove the sample extract (top layer) from the acid using a Pasteur pipet and transfer to a clean vial.

CAUTION: It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.

11.13.4. If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

11.13.5. Dispose of the sulfuric acid waste to the waste sulfuric acid drum for recycling.

11.13.6. Proceed with Section 11.13, sulfur removal.

11.14. Method 3660A, Sulfur Removal with Elemental Mercury.

If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipet, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

Note: Use mercury sparingly in order to minimize exposure and disposal costs.

11.14.1. Transfer 2.0 mL (1L separatory funnel extraction and 3550B/3550C) or 1.5 mL (LVI/RVE and MAE) of the sample extract into a clean concentrator tube or Teflon sealed vial.

11.14.2. Add one to three drops (about 0.25mL) of mercury to the extract vial and seal.

WARNING: Do not use latex or vinyl gloves when working with mercury and organic solvents at the same time.

11.14.3. Vortex for 15-30 seconds.

11.14.4. If black precipitate forms, sulfur was present. Centrifuge, then transfer the supernate to a clean test tube and repeat steps 11.14.2 and 11.14.3. Do this until relatively little precipitate remains. Document if samples require more than five sulfur cleanups. If precipitate is still present after 10 cleanups, re-start with a minimum 5X dilution.

WARNING: Do NOT centrifuge glass tubes containing mercury. Due to the density of the mercury, the test tube could break in the centrifuge.

11.14.5. If no precipitate is present, remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

11.14.6. Rotate the mercury waste out of the laboratory to the Hazardous Waste storage area for labpack disposal.

11.15. Clean-up Cartridge lot verification

11.15.1. Florisil lot verification procedure.

Each lot of Florisil cartridges must be evaluated before use, following this performance check procedure:

11.15.1.1. Combine the following in a clean test tube:

- 1.0mL of a 0.1µg/mL solution of 2,4,5-trichlorophenol solution (in hexane)
- 1.0mL of the 8081 INDA/B Level-5 (Typical OC Pesticide mix @ 20-200µg/mL)

11.15.1.2. Add this mixture to a pre-washed Florisil cartridge. Per the instructions in Section 11.10, elute with 8mL hexane /acetone (90:10). For a blank sample, repeat this step on another cartridge loaded with 2mL of clean hexane. Concentrate the eluates to final volumes of 1.0mL and transfer to glass vials for instrument analysis.

11.15.1.3. Analyze the Florisil check solution and blank, along with an aliquot of the 0.1µg/mL TCP solution per method 8081. Expected recoveries are 80-120% of all pesticide analytes and surrogates with the exception of Endrin Aldehyde, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for waters. Additionally, there should be no indication of

peaks interfering with target compounds, and ≤5% TCP recovery (when peak area is compared between the Florisil check and the native TCP solution).

11.15.2. Styrene Divinylbenzene (SDB) polymer lot verification procedure.

Each lot of SDB cartridges must be evaluated before use, following this performance check procedure:

11.15.2.1. To a pre-washed SDB cartridge, add 2.0mL of the 8081 INDA/B Level-5 (Typical OC Pesticide mix @ 20-200µg/mL)

11.15.2.2. Per the instructions in Section 11.11, elute with 8mL hexane /acetone (90:10). For a blank sample, repeat this step on another cartridge loaded with 2mL of clean hexane. Concentrate the eluates to final volumes of 2.0mL and transfer to glass vials for instrument analysis.

11.15.2.3. Analyze the SDB check and blank, per method 8081. Expected recoveries are 80-120% of all pesticide analytes and surrogates with the exception of Endrin Aldehyde, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for waters. Additionally, there should be no indication of peaks interfering with target compounds.

11.15.3. Graphitized Carbon lot verification procedure.

Each lot of Graphitized Carbon cartridges must be evaluated before use, following this performance check procedure:

11.15.3.1. To a pre-washed Graphitized Carbon cartridge, add 2.0mL of the 8081 INDA/B Level-5 (Typical OC Pesticide mix @ 20-200µg/mL)

11.15.3.2. Per the instructions in 11.12, elute with 8mL hexane /acetone (90:10). For a blank sample, repeat this step on another cartridge loaded with 2mL of clean hexane. Concentrate the eluates to final volumes of 2.0mL and transfer to glass vials for instrument analysis.

11.15.3.3. Analyze the SDB check and blank, per method 8081. Expected recoveries are 80-120% of all pesticide analytes and surrogates with the exception of Endrin Aldehyde, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for waters. Additionally, there should be no indication of peaks interfering with target compounds.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability.) Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracts, extract dilutions, all disposable materials (filters, disposable glassware, etc.) with concentrations of Aroclors greater than 50 ppm. These must receive special handling, shipping and disposal as PCB waste. Contact the Hazardous Waste Technician for specific instructions, as these cannot be disposed in normal waste streams.

15.2. Extracted aqueous/TCLP leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.3. Extracted soil samples, sodium sulfate, glass wool/filter paper, used florisil or other cleanup cartridges, and thimbles contaminated with methylene chloride and acetone from sonication and soxhlet extraction and from sample drying. Pour any excess liquid from the extracted soil samples as outlined below (Section 15.5). Dump these types of wastes into an orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate lab trash collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.4. Used mercury contaminated with sulfur compounds from the sulfur cleanup. Pour the contaminated mercury into a 250-ml plastic bottle labeled for contaminated mercury. When full or after one year, whichever comes first, transfer this jar to the waste collection area for shipment.

15.5. Assorted flammable solvent and methylene chloride waste from soil extraction, various rinses or pre-elutions. Collect the waste solvents in tripours during use. Empty the tripours into a 1liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.6. Used sulfuric acid from the cleanup during 8082/PCB extraction. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III

- Method 3500B, Organic Extraction and Sample Preparation, Revision 2, December 1996
- Method 3510C, Separatory Funnel Liquid Liquid Extraction, Revision 3, December 1996
- Method 3550B, Ultrasonic Extraction, Revision 2, December 1996
- Method 3580A, Waste Dilution, Revision 1, July 1992
- Method 3600C, Cleanup, Revision 3, December 1996
- Method 3620B, Florisil Cleanup, Revision 2, December 1996
- Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II

- Method 3660A, Sulfur Cleanup, Revision 1, July 1992

16.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV

- Method 3500C, Organic Extraction and Sample Preparation, Revision 3, December 2007
- Method 3546, Microwave Extraction, Revision 0, February 2007
- Method 3550C, Ultrasonic Extraction, Revision 3, February 2007

16.4. SOP WS-QA-0025, Glassware Handling Safety

17. METHOD MODIFICATIONS

17.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.2. Aqueous sample volumes are determined by weight.

18. ATTACHMENTS

18.1. Table 1 – Exchange Solvents, Final Volumes, and Aliquot Volumes

18.2. Table 2 – Spiking Volumes

18.3. Table 3 – Spike Solution Components

18.4. Table 4 – Sacramento Method 8081/8082 Extraction Summary

19. REVISION HISTORY

19.1. WS-OP-0002, Revision 4.1, Effective 04/02/2014

19.1.1. Section 2.1.1, revised to read: "This SOP has procedures for extracting both the traditional sample volume (1000mL) as well as reduced sample volumes (250 or 125 mL). The traditional volume is referred to as a "1 L separatory funnel extraction", and the smaller volumes are referred to as "250 mL Limited Volume Initiative/Reduced Volume Extraction" (250 mL LVI/RVE) and "125 mL Limited Volume Initiative/Reduced Volume Extraction (125 mL LVI/RVE) in this SOP."

19.1.2. Section 10.1, changed to read, "On the day of use, measure 2.0 mL of solvent into a 16mL culture tube using a 1.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Seal the culture tube – this is the 'check' or 'reference' vial for cleanup with Florisil. Using the same procedure, create a 10.00mL 'check' vial using a 5.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Record the container lot, solvent, gastight syringe or serological pipet ID, date, and initials of preparer for all check vials in a controlled logbook. The culture tubes containing the sample extracts are then compared against the "check" vial to ensure that the final volume is consistently within the criteria of 10 ± 0.3 or 2 ± 0.06 mL as required."

19.1.3. Section 10.2, changed the frequency of autopipetter verification to daily to reflect changes in WS-PQA-003.

19.1.4. Inserted Section 10.4, "Prior to use, ensure any containers used for quantititative steps (e.g final volume and Florisil) are verified as described in SOP WS-QA-0004."

19.1.5. Section 11.2.4.2, appended to the end of the section, "This must be documented in an NCM, as there may be a low bias to the analysis when the entire container is not use, and the container is therefore not rinsed and the rinsate combined with the extraction solvent."

19.1.6. Inserted Section 11.2.3, "One method for reducing the effects of emulsions is to swirl the funnel after allowing it to rest for the required amount of time, wait approximately a minute, and then drain the extract without collecting the emulsion. After the third (final) extraction and rest period, add a squirt of DCM to identify where the layer of DCM and water is separated and collect only the DCM. Alternatively, on the final pour collect the emulsion and use a second filtration step at KD with glass wool and pipet off excess water."

19.1.7. Appended to the end of the note following Section 11.2.1.4, "Similarly, it is anticipated that only 20 of the 30 mL used (or 10 of 15 mL used) may be recovered on the first shake when extracting smaller water volumes."

19.1.8. Appended to the end of the note following Section 11.2.1.4, "Ensure to minimize the amount of water collected."

19.1.9. Section 11.8.1.11 changed to read, "Remove the KD apparatus from the water bath and allow it to cool and drain. If the level of the extract is above the level of the concentrator tube joint, add a new boiling chip and continue to distill the solvent as necessary. If redistilling, remove the KD apparatus from the water bath and allow it to cool and drain before proceeding to the next step.

19.1.10. Inserted Section 11.8.1.12 to read, "Remove the Snyder column and rinse the inside of the top joint of the K-D flask with a minimum amount of DCM, rotating the flask as necessary to ensure the rinsate covers the sides of the KD flask.

19.1.11. Section 11.8.2.1, inserted at the beginning, "Quantitatively transfer the extract into a calibrated container."

19.1.12. Section 11.8.2.1, changed the second sentence to start, "Alternatively, place the concentrator tube in a warm water bath that…"

19.1.13. Section 11.8.2.3 changed to read, "When the volume is slightly below 10 mL, remove the container/tube from the n-evap bath. Quantitatively transfer the extract into a calibrated container if the extract is still in the concentrator tube. Compare the volume to that of the calibrated reference, and carefully add solvent to make the final volume of 10 mL. Record the lot of the container that extracts are brought to final volume in."

19.1.14. Added references for both 125 mL and 250 mL extraction options under Method 3510C (Separatory Funnel Extraction. Sections updated include: 9.5.1, 11.2.3, 11.2.4.2, 11.2.10, 11.2.1.5, Table 2 (Spiking Volumes), and Table 4.

19.1.15. Tables 2 and 4, the concentration of the technical chlordane standard was

changed from 10 ug/mL to 1.0 ug/mL to permit spiking at a volume consistent with SW846 recommendations.

19.2. WS-OP-0002, Revision 4.0, Effective 7/3/2013

19.2.1. Added warning regarding TSCA requirements for PCB standards at 1000 ppm (Sections 5 and 15)

19.2.2. Removed references to and procedures for continuous liquid-liquid extraction – Section 2 (scope) as well as Section 11 (procedure).

19.2.3. Added references to and procedures for microwave assisted extraction (Method 3546) – Section 2 (Scope) as well as Section 11 (procedure)

19.2.4. Added references to and procedures for Method 3550C – Section 2 (Scope) as well as Section 11 (procedure)

19.2.5. Removed figure 1 (CLLE set-up) as CLLE has been removed.

19.2.6. Added references to SW-846 Update IV methods.

19.2.7. Editorial revisions

19.3. WS-OP-0002, Revision 3.9, Effective 05/10/2013

19.3.1. Updated SOP to conform to current laboratory practices.

19.3.2. Editorial changes.

19.4. WS-OP-0002, Revision 3.8, Effective 04/06/2012

19.4.1. Editorial changes

19.5. WS-OP-0002, Revision 3.7, Effective 07/27/2011

19.5.1. Inserted into Section 11.3.9 and 11.4.6, In pre-approved situations, other solvents (such as Hexane or DCM only) may be used to control co-extracted matrix interference).

19.5.2. Inserted Section 11.7.3, *Styrene Divinylbenzene polymer (SDB-L) sorbant cleanup* (Section 11.10). The SDB-L Styrene-Divinylbenzene polymer SPE tubes (Phenomenex) or DVB SPE tubes (Restek) may be helpful for cleanup of pesticides for analysis by Method 8081A and PCBs by Method 8082. This cleanup should be applied routinely to all extracted soil samples. Use on other matrices is also appropriate if co-extracted contamination is problematic at instrument analysis. This synthetic polymer is used to remove a variety of

unwanted matrix-related compounds.

19.5.3. Inserted Section 11.7.4, *Graphitized Carbon sorbant cleanup* (Section 11.11). The CarboPrep-90 (Restek) or EnviCarb (Supelco) pre-packed cartridges may be helpful for cleanup of pesticides for analysis by Method 8081A and PCBs by Method 8082. This clean-up is not applied routinely as there is approximately 50% recovery of TCX surrogate. Its use should be pre-approved by the department manager. This clean-up removes oils and humic substances.

19.5.4. Added to Section 11.7.5, In pre-approved situations, this acid cleanup can be used for 8081 samples, however, there will be significant loss (or increase) of some compounds, namely: Aldrin, Dieldrin, Endrin, Endosulfan I, Endosulfan II, Endrin Aldehyde, Methoxychlor, Endrin Ketone, and Endosulfan Sulfate

19.5.5. Inserted Section 11.9, Divinylbenzene Styrene Polymer Cartridge Cleanup

19.5.6. Inserted Section 11.10, Graphatized Carbon Cartridge Cleanup

19.5.7. Updated Table entries.

19.5.8. Inserted Appendix 1.

19.5.9. Editorial revisions.

19.6. WS-OP-0002, Revision 3.6 Effective 06/10/2011

19.6.1. Inserted Section 11.4, Method 3540C Extraction of Solids with Soxhlet Apparatus.

19.6.2. Modified Section 11.6.1.3 to: If extract is not proceeding to GPC cleanup, add 50 mL of hexane to the final assembly.

19.6.3. Modified Section 11.6.1.7 to: When the apparent volume of the DCM extract reaches 10-15 mLs, remove the KD apparatus from the water bath and allow it to drain and cool

For samples to be loaded o the Gel Permeation Chromatograph (GPC), adjust the final DCM volume to 10 mL and split this into two, 5 mL portions. Archive one of the 5 mL fractions and GPC the other.

19.6.4. Modified Section 11.6.1.8 to: To solvent exchange to hexane, momentarily remove the Snyder column, add 50 mL of hexane, a new boiling chip, and reattach the Snyder column. Alternatively, pour the exchange solvent into the top of the Snyder column.

19.6.5. Modified Section 11.6.1.9 to: Put the KD assembly back on the water bash

and concentrate the extract to ~5mLs, as described in the previous sections, raising the temperature of the water bath, if necessary, to maintain proper distillation

19.6.6. Inserted new Section 11.6.1.10: Remove the KD apparatus from the water bath and allow it to drain and cool. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If the level of the extract is above the level of the concentrator joint, continue to distill the solvent as necessary. Adjust the extract to 10.0 mL with the solvent last used.

Note: When performing KD on the GPC portion of the extract, bring to a final volume of 5 mL (as the 10 mL extract was split in the previous Section).

19.6.7. Inserted new Section 11.6.1.11. If further concentration is indicated in Table 1, either the micro Snyder column technique or nitrogen evaporation (Section 11.6.2) technique is used to adjust the extract to the final volume required

19.6.8. Editorial revisions.

Table 1					
Exchange Solvents, Final Volumes, and Aliquot amounts					
Type	Exchange Solvent for GPC	Final Volume for GPC	Exchange Solvent for all other clean-ups	Final Volume for LVI/RVE and MAE	Final Volume for 1L separatory funnel and 3550B
No Cleanup	--	--	Hexane	5mL	10mL
GPC Only	DCM	5 mL[1]	--	5mL	5mL
Cartridge clean-up (e.g. Florisil)	--	--	Hexane	5mL - 1.5mL aliquot used	10mL - 2mL aliquot used

[1] Final volume for GPC may be 4 mL if a 2 mL sample loop is used

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

Table 2		
Spiking Volumes		
Solution	**Solution ID**	**Volume (mL)** **1L Sep / LVI / 3550 /3546 / Waste**
Surrogate (8081A, 8082)	0.2 ug/mL DCB/TCX 3/90 P-Surr	1.0 / 0.5/ 2.0 / 1.0/ 2.0
LCS (8081A)	1.0ug/mL Pest Full Spike	0.5 / 0.2 / 0.5 / 0.25 / 0.5
	5ug/mLToxaphene (LCS only)	1.0 / 0.5 / 1.0 / 0.5 / 1.0
	1 ug/mL TChlordane (LCS only)	0.5 / 0.5 / 1.0 / 0.50 / 1
LCS (8082)	2ug/mL PCB Spike Aroclor 1660	1.0 / 0.5 / 1.0 / 0.5 / 1.0

Table 3			
Spike Solution Components			
Solution	**Solvent**	**Compounds**	**Conc. (µg/mL)**
Surrogate	Acetone	Decachlorobiphenyl Tetrachloro-m-xylene	0.2 0.2
Pest Full (8081A)	Acetone	alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Aldrin, Heptachlor, Heptachlor Epoxide, Endosulfan I, Endosulfan II, Endosulfan sulfate, alpha-Chlordane, gamma-Chlordane, Dieldrin, 4,4'-DDE, 4,4'-DDD, 4,4'-DDT, Endrin, Endrin ketone, Endrin aldehyde, Methoxychlor	1ug/mL (for all analytes)
Toxaphene	Acetone	Toxaphene	5
TChlor	Acetone	Technical Chlordane	1
PCB 1660 (8082)	Acetone	Aroclor 1016 Aroclor 1260	2.0 2.0

Table 4							
West Sacramento Method 8081/8082 Extraction Summary							
	Sample Amount	Surrogate	LCS1	Extract Solvent	Extract Solvent Volume	Extraction Time	Comments
1 L separatory funnel extraction	1 L	1.0 mL	See table 3	DCM	3 x 60 mL	2 min shake, 10 min rest	
250 mL LVI/RVE	250 mL	0.5 mL	See table 3		3 x 30 mL		
125 mL LVI/RVE	125 mL	0.5 mL	See table 3		3 x 15 mL		
Leachate (SEPF)	200 mL	1.0 mL	See table 3		3 x 60 mL		Make the 200mL to 1L with DI water before extracting
Solid (Sonication)	30 g	2.0 mL	See table 3	1:1 DCM /Ace	3 x 100mL	3min	
Waste Dilution	1 g	2.0 mL	See table 3	Hex	10mL	NA	Don't concentrate.
Biologics (Sonication)	10 g	20 mL	See table 3	1:1 DCM /Ace	3 x 100mL	3 min	
Solid (Microwave)	15g	1.0mL	See table 3	1:1 Hex /Ace	25mL	25min	

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Title: Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW-846 3500 Series and 3600 Series Methods for Analysis by Methods 8015B, CA-LUFT, NW-TPH, and AK 102/103 [Methods 8015B and AK 102/103]

Approvals (Signature/Date):

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1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, solid and waste matrices for analysis by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA).

1.1. Extraction procedures for the TPH-Diesel determinative methods are covered.

1.2. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

1.3. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction: A measured volume of sample is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.1.1. This method is subdivided into two extraction methods: typically a 1000mL nominal sample volume extracted in 2L separatory funnels (henceforth referred to as a 1L separatory funnel extraction in this SOP), and typically a 125mL sample volume extracted in a 250mL separatory funnels (henceforth referred to as a Large Volume Injection/Reduced Volume Initiative or LVI/RVE in this SOP).

2.2. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent-extracted with methylene chloride three times using an ultrasonic horn.

WARNING: Hearing protection is required when sonicating samples.

2.3. Soxhlet Extraction

A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing methylene chloride for 16-24 hours.

2.4. Waste Dilution

A 1 g sample aliquot is diluted to 10 mL with methylene chloride.

2.5. Microwave Extraction

A measured weight of sample, typically 15g (10 g for biologics), is extracted with Methylene Chloride:Acetone 1:1 (DCM:ACE 1:1).

2.6. Cleanup and Concentration

Procedures are presented for removing interferents from sample extracts, and for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3. To prevent analyte loss, extracts which are not immediately concentrated by KD must be protected from light during storage. Extracts may be either refrigerated or stored in an opaque container.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Microwave ovens used for extraction or digestion of samples can create

elevated pressure in the extraction/digestion containers. Microwaves used for these processes must be equipped with an automated vapor sensor and shutoff system that automatically shuts down the digestion/extraction process when vapor pressure reaches pre-specified levels. Users must follow procedures in the microwave operators manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.5. When Soxhlet extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.1.6. Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

5.1.7. Assembly and disassembly of glassware creates a risk of breakage and cuts.

All staff members shall wear Kevlar® or similar cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.8. All glassware must be inspected before use. Chipped, cracked or broken glassware must be removed from service immediately, especially glass separatory funnels. This glassware must either be sent out for repair or discarded.

5.1.9. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.1.10. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the process may be performed behind a closed fume hood sash. After each of the three extraction cycles, ensure that the stoppers are removed from the top of the separatory funnels.

5.1.11. The kiln used to clean glassware creates an extreme risk of severe burns. Allow sufficient time for the kiln to cool before opening the doors. Ensure that appropriate heat protective gloves are worn when working with freshly kilned glassware. Other surfaces in the kiln may remain hot; so exercise caution when reaching into the kiln.

5.1.12. All personnel are to ensure liquid-liquid area is clear of unnecessary items. Heating mantles used with liquid-liquid extractions generate temperatures that could ignite some materials that come in contact with the heating mantles.

5.1.13. Ensure that all solvents are kept away from liquid-liquid extractors. Increased temperatures near solvents can cause the pressure in the containers to increase.

5.1.14. Ensure that all boiling flasks have cooled to room temperature before disconnecting liquid-liquid bodies from boiling flasks to prevent any burns.

5.1.15. Ultrasonic disrupters produce high intensity noise and must be used in an area with adequate noise protection. Hearing protection must be worn when operating sonicators.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain, and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned with soap and water, rinsed with water and kilned in an oven at 400°C for at least 2 hours. Refer to SOP WS-OP-0011 for details of glassware cleaning. Kilned glassware does not require solvent rinsing before use.

WARNING: Heat resistant gloves must be used when handling kilned glassware.

6.2. Equipment and supplies for extraction procedures.

6.2.1. Separatory funnel, 2L (Glass)

6.2.2. Separatory funnel, 500mL (Glass)

6.2.3. Separatory funnel rotator with timer

6.2.4. Balance: >1400 g capacity, accurate ±0.1 g

6.2.5. pH indicator paper, wide-range: covers extraction pH

6.2.6. Graduated cylinder: 1 liter. (Other sizes may be used)

6.2.7. Erlenmeyer flask, beaker, or French square jar: 125 & 300 mL (other sizes optional)

6.2.8. Solvent dispenser pump or 100 mL graduated cylinder

6.2.9. Round or flat bottom flask: 250, 500 mL or 1 L

6.2.10. Boiling chips: contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).

6.2.11. Cooling condensers

6.2.12. Heating mantle: Rheostat controlled, with timer

6.2.13. Beakers: 250 & 400 mL, graduated

6.2.14. Balance: >100 g capacity, accurate to ± 0.01g

6.2.15. Soxhlet extractor

6.2.16. Sonicator (at least 300 watts), with sonicator horn, 3/4 inch

6.2.17. Kuderna-Danish (KD) apparatus: 500 mL flask, with 10mL concentrator tube, and 3-ball macro Snyder column

6.2.18. Locally assembled solvent vapor condensing/recovery system for use with 6.2.17 above.

6.2.19. Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.

6.2.20. Nitrogen blowdown apparatus,

6.2.21. Culture tubes: 10 mL, 16 mm x100 mm

6.2.22. Syringe: 1 mL

6.2.23. Glass wool
To ensure glass wool is free of contaminants, it is necessary to soxhlet rinse glass wool in methylene chloride for 18 hours.

6.2.24. Glass funnel: 75 X 75 mm

6.2.25. Disposable pipets

6.2.26. Aluminum foil

6.2.27. Paper towels

6.2.28. Pressure rotor and pressure reactor

6.2.29. Extraction vessel and extraction vessel cover

6.2.30. Glass inserts

6.2.31. Microwave – Milestone Ethos EX

6.3. Equipment and Supplies for Cleanup Procedures

6.3.1. Mechanical shaker or mixer: Vortex Genie or equivalent.

6.3.2. Disposable pipets

6.3.3. Culture tubes: 10 mL, 16 mm x100 mm

7. REAGENTS AND STANDARDS

7.1. All reagents must be ACS reagent grade or better unless otherwise specified.

7.1.1. Sodium hydroxide solution, 10 N

7.1.2. Sulfuric acid (H_2SO_4), concentrated: reagent grade

7.1.3. Sulfuric acid (1:1): carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.

WARNING: Always add concentrated acids or bases to water, never add water to the concentrated acid or base. Due to the reaction between acid and water, add the acid slowly to water to prevent breaking of the mixing vessel

7.1.4. Organic free reagent water (deionized water). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks, in accordance with SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water."

7.1.5. Sodium sulfate (Na_2SO_4), granular, anhydrous, reagent grade: Purify by heating at 400°C for a minimum of two hours.

7.1.6. Extraction/exchange solvents: methylene chloride, acetone.

7.1.7. Acetone: used for cleaning.

7.1.8. Nitrogen, reagent grade.

7.1.9. Silica gel.

7.2. Standards

7.2.1. Source Standards

Source standards are purchased as certified solutions or prepared from neats. Semivolatile source standards are stored at ≤ 6°C. All stock standards must be protected from light.

7.2.1.1. Source standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Standards must be allowed to come to room temperature before use.

7.2.1.2. Expired standards must be rotated out of the lab to the Hazardous Waste storage area.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 1. Surrogate spiking standards are prepared as dilutions of source standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the Matrix Spike (MS) and the Laboratory Control Sample (LCS). Prepare MS/MSD/LCS spiking standards at the concentrations listed in Table 2. Spiking standards are purchased or prepared as dilutions of stock standards. Spiking solutions must be refrigerated and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved (exception: Samples for Alaska methods AK102 & AK103. See Section 8.3.1).

8.2. Samples are stored at ≤ 6°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples. Aqueous samples for California LUFT may be extracted within 14 days with client approval. Holding time for aqueous samples for Alaska methods AK102 & AK103 is 14 days when preserved to pH < 2.

8.3.2. For TCLP leachates, extraction is initiated within seven days from when the leaching procedure is completed.

8.3.3. Analysis of the extracts must be completed within forty days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a MB, an LCS and a MS/MSD. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients request specific samples for MS/MSD, the batch may contain multiple MS/MSD. See policy WS-PQA-003 for further definition of the batch.

9.2. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated

according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented using the NCM program in TALS.

9.3. Method Blank

A MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.3.1. Aqueous MB use 1000mL for 1L separatory funnel extraction or 250mL for LVI/RVE of organic free reagent water (deionized water) spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.2. Solid MB use 30g of kilned sodium sulfate for 3550B/C and 15g of kilned sodium sulfate for 3546 spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.3. TCLP MB use 200 mL of leachate fluid spiked with the surrogate. The MB goes through the entire analytical procedure, including any cleanup steps.

9.3.4. Waste MB use methylene chloride spiked with the surrogates. The MB goes through the entire analytical procedure, including any cleanup steps.

9.4. Laboratory Control Sample (LCS)

LCS is a well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.4.1. The LCS is made up in the same way as the Method Blank (See Sections 9.3.1 through 9.3.4) but is spiked with the LCS standard and the surrogates. There is no leachate LCS for this method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, MB, LCS and MS/MSD is spiked with surrogate

standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS is an environmental sample to which known concentrations of target analytes have been added. A MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. The MS and MSD are spiked with the LCS standard and the surrogate.

9.7. Laboratory Control Sample Duplicate (LCSD)

A laboratory control sample duplicate (LCSD) may be substituted for a MS/MSD if there is insufficient sample available to process a MS/MSD, if precision data is required by the client or program. Certain programs, such as Alaska or Arizona, require an LCSD in addition to LCS/MS/MSD. In the cases where an LCSD is processed, the LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented on an NCM.

Samples for compliance within the State of Alaska (AK102 and AK103) require an LCS/LCSD pair with every batch.

9.8. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.9. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.10. West Sacramento QC Program

Further details of QC and corrective action guidelines are presented in the Sacramento QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. On the day of use, measure either 3.00 mL or 2.00 mL of methylene into a 16 mL culture tube using a 1 mL syringe that is calibrated in accordance with SOP WS-QA-0004. Seal the culture tube – this is the ‘check’ or ‘reference’ vial. The culture tubes containing the sample extracts are then compared against the “check” vial to ensure that the final volume is consistently 3.0 ± 0.01 mL or 2 ± 0.01 mL depending on the required final volume.

10.2. On the day of used, verify any auto-pipettors to be used in accordance with SOP WS-QA-0004.

10.3. On the day of use, verify any balances to be used in accordance with SOP WS-QA-0041.

10.4. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed, or annually, whichever is sooner.

10.4.1. Newer models of sonicators may be self-tuning. Read the manufacturer's directions prior to operating this equipment.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Method 3510C, Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and LCS spiking standards from refrigerator and allow standards to warm to room temperature.

11.2.2. If the sample bottle contains a layer of sediment greater than approximately ½" in depth, contact the department manager and the project manager to determine how to proceed with the sample extraction. Options may include the following:

a) Decanting and extracting only the liquid fraction in a separatory funnel before adding the surrogate and/or spike solutions.
b) Adding the surrogate and/or spike solutions to the sample with the sediment still in the original sample container.
c) Processing the aqueous portion as described in a) along with creating a new sample id for the soil matrix. The sample bottle would then be rinsed with 60 mL of DCM, spiked with an appropriate volume of surrogate solution and shaken for 3 minutes. The addition of 60 mL of DCM will be repeated 2 more times and

all solvents combined to create a second extract for the sample that is to be added to the extraction batch of water samples

NOTE: In the case of option "b", after adding the appropriate surrogates and/or spikes, the sample would be gently swirled for mixing purposes and then gently poured into the separatory funnel, thus minimizing the amount of sediment transferred to the extraction vessel during the process. The original sample container would then be rinsed with 60 mL of DCM (1 L extraction) or 30 mL (LVI/RVE extraction). This DCM rinseate would then be poured into the extraction vessel.

11.2.3. The normal sample volume is 1 liter for 1L separatory funnel extractions and 250 mL for LVI/RVE. Other sample volumes may be used to obtain specific reporting limits, or accommodate dirty samples and reduced sample volumes, diluted to 1 L or 250 mL with organic free reagent water (deionized water).

11.2.4. Measure the initial sample pH with wide-range pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record on the extraction benchsheet. If the sample is a leachate, e.g. TCLP, compare the current pH against the leachate log and note on the benchsheet if there is any discrepancy.

11.2.5. Obtain sample weight(s) to ± 0.1 g. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. This may be done in two ways depending if a dilution is necessary.

11.2.5.1. For samples not requiring a dilution weigh the gross container on a tared balance and after extraction has been initiated weigh the tared container on a tared balance and use the difference as the sample weight.

11.2.5.2. For samples requiring a dilution, subsample the desired aliquot amount in accordance with SOP-QA-0018 into a tared container. Dilute to 1L nominal for 1L separatory funnel extraction and 250mL nominal for LVI/RVE with organic free reagent water (deionized water).

11.2.5.3. For TCLP samples, use 200mL of leachate and dilute to 1L in organic free reagent water.

Note: *Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor*

accuracy. However, either method may be necessary for specific client programs.

11.2.6. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.2.7. For a TCLP batch prepare a MB and LCS as specified in Section 9 of this SOP. In addition, prepare a TCLP MB by measuring 200 mL of buffer solution used in the leaching procedure and dilute to 1 L nominal using the methods described in Section 11.2.5.

11.2.8. After samples have warmed to room temperature, add the surrogate standard to all samples and the LCS solution to the necessary samples as described in Section 9 and Table 2 of this SOP. Return spiking solution to the refrigerator as soon as possible after spiking. .

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer part of the sample to the separatory funnel and then add the spike to the bottle.

11.2.9. Rinse the sample bottle with 60mL methylene chloride for 1L separatory funnel extraction and 30mL for LVI/RVE and transfer to the 2L glass or Teflon separatory funnel for 1L separatory funnel extraction and 250mL glass or Teflon separatory funnel for LVI/RVE.

11.2.10. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. As described in Section 5.1.10, Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle.

WARNING: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.11. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (at least 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. The emulsion is considered broken when recovery of <80% of the methylene chloride* is achieved

**15 – 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 40 mL from the first shake and still be acceptable for 1L separatory funnel extraction. Subsequent shakes should recover at least 50 mL of solvent for 1L separatory funnel extractions. Apply the same ratios for LVI/RVE.*

11.2.12. Fill a funnel with anhydrous kilned sodium sulfate (10-15 g for LVI/RVE, nearly a full funnel for 1L separatory funnel extraction). The funnel can be plugged with a small amount of glass wool to hold the sodium sulfate. Rinse the funnel with methylene chloride and discard the rinseate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate prior to the concentration step.

11.2.13. Repeat the extraction process two more times using fresh 60 mL portions of solvent for 1L separatory funnel extraction and 30mL for LVI/RVE, combining the three solvent extracts in the collection container.

11.2.14. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.15. Dispose of solvent and water remaining in the extractor into the LLE (liquid/liquid extraction) waste drum. Waste methylene chloride goes to the DCM drum for recycling.

11.2.16. Cap the collection container if the extract is not concentrated immediately. Refer to Section 11.6 for concentration and Section 11.9 for cleanup.

11.3. Method 3550B and 3550C, Solid Extraction by Sonication

11.3.1. Remove surrogate and matrix spiking solutions from refrigerator and allow them to warm to room temperature.

11.3.2. Decant and discard any water layer on a sediment/soil sample, unless otherwise specified by the client. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator). Document if a water layer was

discarded.

11.3.3. Weigh 30 g of sample ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0.1 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate for the method blank and LCS.

11.3.4. Mix weighed sample with a spatula or blending rotor, adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced).

11.3.5. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.3.6. Add the surrogate and spike standard to the necessary samples as described in section 9 and table 2 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.3.7. Immediately add 100 mL of methylene chloride to the beaker.

Note: Steps 11.3.3 – 11.3.7 should be performed rapidly to avoid loss of the more volatile extractables.

11.3.8. Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pour beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horn with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

11.3.9. Place the bottom surface of the sonicator disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.3.10. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: Do **not** use **Microtip** probe.

WARNING: Hearing protection is required when sonicating samples.

11.3.11. Decant extracts after each sonication into a new 16 oz French square jar.

Note: Do not use old kilned French square jars as collection vessels. These are a potential source of contamination.

11.3.12. Take extra care when decanting/transferring the solvent to the collection vessel to minimize, as much as possible, the amount of soil and sodium sulfate that is transferred along with the solvent. After the final sonication decant the solvent into the collection vessel and leave as much of the sodium sulfate and soil as possible in the original jar. Cap the collection jar. If concentration of the extracts is not going to be performed on the same day, store the extracts in a flammable refrigerator that is set at 2-6 °C.

11.3.13. Repeat steps 11.3.10 – 11.3.12 two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication.

11.3.14. Cap the collection container. Refer to Section 11.6 for concentration and Section 11.9 for cleanup.

11.4. Method 3546, Solid Extraction by Microwave

11.4.1. Remove surrogate and matrix spiking solutions from refrigerator and allow standards to warm to room temperature.

11.4.2. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.3. Weigh 15g of sample (10g for biologics) ± 1.0g into a clean (soap and water followed by DCM rinsing) extraction vessel. Record the weight to the nearest 0.01g in the appropriate column on the benchsheet.

11.4.3.1. For visually dirty samples, it may be necessary to weigh the sample into a glass insert to be placed into an extraction vessel. Contact your supervisor to determine if this is the case.

11.4.4. Mix weighed sample with a spatula. If clay clumps or other sample aggregation is evident, add kilned sodium sulfate (not to exceed 5 g) as needed to facilitation disaggregation and mix as necessary.

11.4.5. Add the surrogate and spike standard to the necessary samples as described in section 9 and table 2 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.4.6. Add the surrogate spiking solution to each sample, MB, LCS, and MS/MSD. Add the appropriate matrix spiking solution to each MS/MSD and LCS. Record spiking volumes and standard numbers on the benchsheet. Return

spiking solutions promptly to refrigerator. Immediately add 25 mL of DCM:ACE 1:1 to extraction vessels.

Note: Steps 11.4.3 - 11.4.6 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.7. Add DCM-rinsed flared extraction vessel covers to each extraction vessel containing sample and QC aliquots.

11.4.7.1. It is important that each cover fits snugly to ensure a proper seal. The cover should not slide easily or loosely inside the extraction vessel, but should require some finger pressure to insert firmly. A cover flaring tool should be used.

11.4.7.2. For the visually wettest sample, add the thermowell liner into the extraction vessel cover to create the representative sample that the ATC temperature sensor can be inserted into.

11.4.8. Place each extraction vessel into a pressure reactor. Screw on the pressure cap/safety lid. The pressure cap should be hand tightened until the sealing valve is flush with the top of the cap.

11.4.8.1. For the representative sample created in 11.4.7.2, add the protection foil and appropriate safety lid.

11.4.9. Place all the extraction vessels into the rotor so that the pressure-release valves are facing outside of the rotor on the outside ring and inside toward the center on the inside ring.

WARNING: Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Follow procedures in the operators manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.

11.4.9.1. Place the rotor in the microwave oven and insert the ATC temperature sensor into the representative sample (11.4.7.2 and 11.4.8.1).

11.4.10. Close the microwave oven and start the appropriate extraction profile.

11.4.10.1. A 10 minute ramp from 25°C to 100 °C , hold for 25 minutes at 100°C , followed by a 15 minute cooldown to 25°C.

11.4.11. After the extraction period and cooldown, pressure reactors should be at ambient temperature prior to removal from the rotor and opening.

11.4.12. Proceed to Section 11.6 concentration

11.4.12.1. Discard any glass inserts. To clean extraction vessels and pressure cap/safety lid, use soap and water followed by a DCM solvent rinse. For visually dirty samples and/or samples with an odor, run the vessels with a cleaning cycle by repeating 11.4.6 through 11.4.11 and discarding the rinsate. Use only solvent to rinse the probe. Allow to air dry prior to use.

11.5. Method 3580A, Waste Dilution

11.5.1. This method is used for materials that are soluble in an organic solvent.

11.5.2. Remove surrogate and matrix spiking solutions from refrigerator and allow them to warm to room temperature.

11.5.3. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest 0.01 g.

11.5.4. Prepare a MB, LCS, and LCSD for each batch as specific in Section 9 of this SOP.

11.5.5. Add approximately 3mL of methylene chloride. Add the surrogate and spike standard to the necessary samples as described in section 9 and table 2 of this SOP. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.5.6. Exchange to methylene chloride by bringing the sample to approximately 13mL and then under nitrogen blowing back to approximately 4mL.

11.5.7. Compare the volume to that of the calibrated reference container, and carefully add methylene chloride to make the final volume of 10 mL. Ensure to record the lot of the container that extracts are brought to final volume in.

11.5.8. Add approximately 2g of sodium sulfate to the samples. Cap and shake for 2 minutes.

11.5.9. Remove an (approximately 3mL) aliquot for analysis. The excess in the culture tube is stored in a refrigerator at 0 -6°C for a minimum of 30 days (can be used for additional re-analysis as needed) and disposed of in accordance with section 15 of this SOP.

11.6. Concentration

According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Below are the “normal” final volumes for tests covered by this SOP:

Test	Final Volume	Split?
Normal 3550B/1LS	3.0mL	No
NWTPH-Dx 3550B/1LS	5.0mL	No
Si Gel Cleaned 3550B/C/1LS	3.0mL	1.5mL for cleaning, 1.5mL for archive.
Hi-Level 3550B/C/1LS	15mL	No
AK102/103 3550B/1LS	3.0 mL	No
MAE & LVI/RVE	2.0mL	No

11.7. Kuderna-Danish (KD) Method

11.7.1. Loosely plug the stem of a 75mm x 75mm glass funnel with glass wool. (Note: Only use glass wool that has been soxhlet rinsed with DCM for 18 hours.) For solid samples and LVI/RVE add 5-10g of kilned anhydrous sodium sulfate to the funnel cup. For 1L separatory funnel extraction, add anhydrous sodium sulfate to nearly fill up the funnel cup. RINSE the entire funnel cup set-up with DCM until the sodium sulfate is completely immersed by the DCM. This is imperative to remove possible contaminants.

11.7.2. Place the prepared funnel cup into a ring stand over the KD flask. Decant and filter extracts through the prepared funnel into the KD apparatus. Make sure to rinse the French square jar 3 times with DCM, decanting each rinse through the funnel cup into the KD flask. This procedure helps to ensure adequate analyte recovery.

11.7.3. Assemble a Kuderna-Danish concentrator as shown in Figure 2 by attaching a 10 mL concentrator tube to the 500 mL KD flask. Transfer the sample to the KD flask. Add one or two DCM rinsed boiling chips and the extract to be concentrated to the KD flask, and attach a three ball Snyder column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly). Attach the solvent vapor recovery device to the top of the Snyder column.

11.7.4. Place the KD apparatus on a water bath (80-85ºC) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

11.7.5. Concentrate to about 6 mL.

Note: It is very important not to concentrate to dryness, as analytes will be lost.

11.7.6. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again,

allow the KD flask to cool for a minimum of 10 minutes. Proceed with Section 11.7.7, unless cleanups are requested. For cleanups, proceed to Section 11.9.

11.7.7. Quantitatively transfer the extract to a 16 mL test tube. Pipet the extract into the tube, then rinse the KD tube with 1-2 mL of methylene chloride, and add the rinsate to the test tube. Repeat twice with fresh portions of solvent. If the volume in the tube is greater than the final volume required, proceed to Section 11.8. Otherwise, adjust to the final volume by adding methylene chloride.

11.8. Nitrogen Evaporation to Final Concentration

11.8.1. Place the tube in a water bath that is no warmer than 35°C and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

11.8.2. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 3.0 mL.

11.8.3. When the volume is slightly below 3.0 mL, remove the test tube from the n-evap bath. Compare the volume to that of the calibrated reference, and carefully add solvent to make the final volume.

Note: It is very important not to concentrate to dryness, as analytes will be lost.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.8.4. If requested, proceed to the cleanup steps. Otherwise, extracts are ready for analysis.

11.9. Cleanup Techniques

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/ or loss of detector sensitivity. In general, these cleanups are applied only when the client requests them. As of this writing, no cleanups are performed on LVI/RVE and MAE extracts.

Silica Gel cleanup (Section 11.10) is used to remove interferents of biological origin from the extract.

Northwest cleanup (Section 11.11) is an acid and silica gel cleanup, applied at client request to comply with requirements for the states of Oregon and Washington.

WARNING: Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

11.10. Silica Gel Cleanup

11.10.1. Adjust the extract volume to 3.0 mL with methylene chloride.

11.10.2. 1.5 ml is silica gel cleaned, 1.5 mL is archived.

11.10.3. To a "Monstr-pette" (Chase Scientific Glass 5.75 inch) plugged with a small amount of glass wool, add ~1.5 g activated silica gel (baked 8 hours at 150-160 deg C). Top with a small layer of kilned sodium sulfate.

Note: If a "Monstr-pette" is not available, a 5 mL graduated pipette may be used.

11.10.4. Rinse column by filling to the top with methylene chloride and allowing the methylene chloride to collect in a 16 mL test tube. Repeat for a total of 3 rinses. Positive pressure using a pipette bulb may be used. Discard methylene chloride rinse and test tubes.

11.10.5. Add 1.5 mL sample to the column, and collect in a clean 16 mL test tube. Elute sample with ~6 mL methylene chloride. A pipette bulb may be used to speed the sample through the column. Adjust the volume to 1.5 mL under nitrogen (See Section 11.8.)

11.10.6. All samples including the batch QC (MB/LCS/LCSD) and field QC (MS/MSD) are cleaned.

11.11. Northwest Cleanup

11.11.1. Adjust the extract volume to 5.0 mL with methylene chloride.

11.11.2. Add approximately 0.5 mL of concentrated sulfuric acid to the extract and cap.

WARNING: There must be no water present in the extract or the reaction may shatter the sample container.

11.11.3. Shake or vortex for about one minute and allow it to settle. (Centrifuge if necessary)

11.11.4. Remove the sample extract (top layer) from the acid using a Pasteur pipet and transfer to a clean culture tube.

CAUTION: It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.

11.11.5. Add approximately 0.2 g silica gel to the extract.

11.11.6. Shake or vortex for about one minute and allow it to settle. (Centrifuge if necessary)

11.11.7. Remove the sample extract from the silica gel using a Pasteur pipette and transfer to a clean culture tube.

11.11.8. Repeat steps 11.11.2 through 11.11.7.

11.11.9. Mark the top and bottom meniscus on the tube, and transfer to the instrument area.

11.11.10. Dispose of the sulfuric acid waste to the waste sulfuric acid drum for recycling.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The

concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/TCLP leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples, thimbles, used sodium sulfate, glass wool or filter paper contaminated with methylene chloride and acetone. Dump the extracted soil and thimbles into an orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Assorted flammable solvents and methylene chloride waste from various rinses. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift,

whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.4. Miscellaneous disposable glassware and silica gel contaminated with acids, caustics, solvents and sample residue. Dump the material into a yellow contaminated lab trash. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.5. Used sulfuric acid from the Northwest cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

15.6. Expired standards must be rotated out of the laboratory for disposal to the vial waste stream.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III

Method 3500B, Organic Extraction and Sample Preparation, Revision 2, December 1996

Method 3510C, Separatory Funnel Liquid Liquid Extraction, Revision 3, December 1996

Method 3540C, Soxhlet Extraction, Revision 3, December 1996

Method 3550B, Ultrasonic Extraction, Revision 2, December 1996

Method 3580A, Waste Dilution, Revision 1, July 1992

Method 3600C, Cleanup, Revision 3, December 1996

Method 3630C, Silica Gel Cleanup, Revision 3, December 1996

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV

Method 3500C, Organic Extraction and Sample Preparation, Revision 3, December 2007

Method 3546, Microwave Extraction, Revision 0, February 2007

Method 3550C, Ultrasonic Extraction, Revision 3, February 2007

16.3. Leaking Underground Fuel Tank Field Manual, State of California, October, 1989

16.4. Underground Storage Tanks Procedures Manual, Alaska Department of Environmental Conservation, November 7, 2002; Appendix D

16.5. Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997

16.6. Alaska Department of Environmental Conservation, April 8, 2002; Methods AK102.0 and AK103.0

16.7. Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997, Method NWTPH-Dx

16.8. EPA Memorandum (Dated August 5, 2010) Regarding "Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods"

17. METHOD MODIFICATIONS

17.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.2. Aqueous sample volumes are determined by weight.

17.3. Spiking methods for 3550C is performed after mixing with drying agents, as per the EPA surrogate spiking memo (Section 16.8 above).

18. ATTACHMENTS

18.1. Table 1- Standard Reagent Mixtures/Sources

18.2. Table 2 – Spiking Table

18.3. Table 3 – Sacramento Method 8015B Extraction Summary

19. REVISION HISTORY

19.1. WS-OP-0004, Revision 5.2, Effective 5/27/2015

19.1.1. Section 2.5 – replaced methylene chloride with methylene chloride:acetone 1:1.

19.1.2. Section 9.3.2 – replaced Ottawa sand with kilned sodium sulfate.

19.1.3. Replaced Section 11.4 (Method 3550C, Solid Extraction by Sonication) with procedure (Method 3546, Solid Extraction by Microwave).

19.1.4. Removed Section 11.5 (Method 3540C, Extraction of Solids with Soxhlet Apparatus), and renumbered accordingly.

19.1.5. Added Section 16.8 – "EPA Memorandum (Dated August 5, 2010) Regarding "Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods"

19.1.6. Added Section 17.3 – "Spiking methods for 3550C is performed after mixing with drying agents, as per the EPA surrogate spiking memo (Section 16.8 above)."

19.1.7. Editorial changes.

19.2. WS-OP-0004, Revision 5.1, Effective 06/27/2014

19.2.1. Revised Section 9.4.1 to read – "The LCS is made up in the same way as the Method Blank (See Sections 9.3.1 through 9.3.4) but is spiked with the LCS standard and the surrogates. There is no leachate LCS for this method."

19.2.2. Editorial changes.

19.3. WS-OP-0004, Revision 5.0, Effective 08/30/2013

19.3.1. Added reference and procedures for reduced volume separatory funnel extraction (Method 3510C) – Section 2 (Scope) as well as Section 11 (procedure)

19.3.2. Removed reference to and procedures for continuous liquid-liquid extraction. – Section 2 (Scope) as well as Section 11 (procedure)

19.3.3. Added reference and procedures for microwave assisted extraction (Method 3546) – Section 2 (Scope) as well as Section 11 (procedure)

19.3.4. Added reference and procedures for Method 3550B – Section 2 (Scope) as well as Section 11 (procedure)

19.3.5. Section 9.7 – Added remark regarding the LCSD requirement for Alaska work.

19.3.6. Removed figure 1 (CLLE set-up) as CLLE has been removed.

19.3.7. Added references to SW-846 Update IV methods.

19.3.8. Editorial Revisions

19.4. WS-OP-0004, Revision 4.3, Effective 12/14/2012

19.4.1. Removed reference to filter paper as a plug for sodium sulfate funnels. Only glass wool is used as filter paper causes contamination.

19.4.2. Editorial changes.

19.5. WS-OP-0004, Revision 4.2, Effective 2/29/2010

19.5.1. Added Section 1.3: When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPPP Implementation" must be checked and incorporated.

19.5.2. Added Sections 11.2.3.1 and 11.3.1.1: If the sample bottle contains a layer of sediment greater than approximately ½" in depth, contact the department manager and the project manager to determine how to proceed with the sample extraction. Options may include the following:

a) Decanting and extracting only the liquid fraction in a separatory funnel before adding the surrogate and/or spike solutions.
b) Adding the surrogate and/or spike solutions to the sample with the sediment still in the original sample container.
c) Processing the aqueous portion as described in a) along with creating a new sample id for the soil matrix. The sample bottle would then be rinsed with 60 mL of DCM, spiked with an appropriate volume of surrogate solution and shaken for 3 minutes. The addition of 60 mL of DCM will be repeated 2 more times and all solvents combined to create a second extract for the sample that is to be added to the extraction batch of water samples

NOTE: In the case of option "b", after adding the appropriate surrogates/spikes, the sample would be gently swirled for mixing purposes and then gently poured into the separatory funnel, thus minimizing the amount of sediment transferred to the extraction v vessel during the process. The original sample container would then be rinsed with 60 mL of DCM. This DCM rinseate would then be poured into the extraction vessel.

19.6. WS-OP-0004, Revision 4.1, Effective 1/15/2010

19.6.1. Updated Sections 11.4 and 11.8 to reflect current extract decanting procedures.

19.6.2. Inserted Section 11.4.8 "Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pod beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horn with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

19.6.3. Changed all surrogate references in Section 11 from OTP to OTP/Triacontane.

Table 1						
Standard Reagent Mixtures/Sources						
Mix Name	**Analyte Name**	**Source Concentration**	**Source Amount**	**Solvent**	**Final Volume**	**Final Conc. µg/mL**
TPH Surrogate	o-Terphenyl	8000	2.5mL	Acetone	500mL	40
Triacontane Surrogate	n-Triacontane-d62	5000	2mL	80:20 ACE:HEX	200mL	50
TPH Spike	Composite # 2 Diesel	50000	2.4mL	Acetone	200mL	600
Motor Oil Spike	Motor Oil	50000	3.6mL	Acetone	100mL	1800

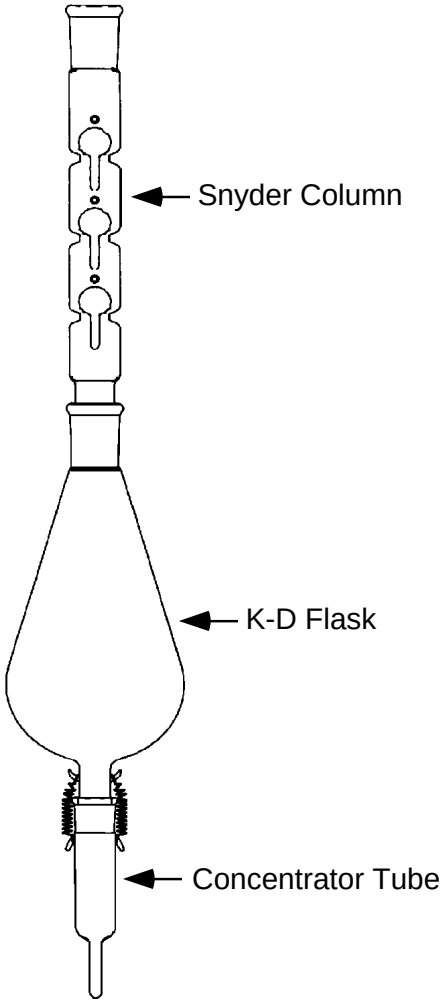
Table 2					
Spiking Table					
Method	**Test**	**Surrogate Volume**	**Name**	**Spike Volume**	**Name**
1L Separatory	8015/AK102	1mL	TPH Surrogate	0.5mL	TPH Spike
	AK103	1mL	TPH Surrogate	0.5mL	Motor Oil Spike
		1mL	Triacontane Surrogate		
	NWTPH	1.5mL	TPH Surrogate	0.75mL	TPH Spike
LVI/RVE	8015/AK102	0.5mL	TPH Surrogate	0.5mL	TPH Spike
	AK103	0.5mL	TPH Surrogate	0.5mL	Motor Oil Spike
		0.5mL	Triacontane Surrogate		
3550B/C	8015/AK102	1mL	TPH Surrogate	0.5mL	TPH Spike
	AK103	1mL	TPH Surrogate	0.5mL	Motor Oil Spike
		1mL	Triacontane Surrogate		
	NWTPH	1.5mL	TPH Surrogate	0.75mL	TPH Spike
3546	8015/AK102	0.5mL	TPH Surrogate	0.5mL	TPH Spike
	AK103	0.5mL	TPH Surrogate	0.5mL	Motor Oil Spike
		0.5mL	Triacontane Surrogate		
3580	8015	3mL	TPH Surrogate	1.5mL	TPH Spike and/or Motor Oil Spike (see analyte list)

***If Jet Fuel (JPA, JP4, or JP8) is an analyte of concern, consult the department manager about concentration/volumes to be spiked.**

Table 3				
Extraction parameters by extraction type:				
Method	**Sample Amount**	**Extract Solvent Volume**	**Extraction Time**	**Comments**
Aqueous (1LS)	1 L	3 x 60 mL DCM	2 min shake, 10 min rest	
Solid (Sonc)	30 g	3 x 100 mL DCM	3 min	
Waste Dilution	1 g	10 mL DCM	NA	Don't concentrate.
Wipes	1 wipe	3 x 100 mL DCM	16-24 hours	
Aqueous (LVI/RVE)	250mL	3 x 30 mL DCM	2 min shake, 10 min rest	
Solid (MAE)	15g	25 mL DCM:ACE 1:1	25 minute	

Note: 1LS – 1 Liter Separatory Funnel; LVI/RVE –Large Volume; MAE – Microwave; Sonc - Sonication

Figure 1 — KD Setup (See Section 11.7 for description)



Title: Determination of All Types of Residue in Water, Wastes, and Solid Samples [Standard Methods 2540B-1997, 2540C-1997, 2540D-1997, and 2540G-1997]

Approvals (Signature/Date):

Thep Phomsopha — Date 11/14/13 Inorganics Supervisor	Joe Schairer — Date 11/15/13 Health & Safety Manager / Coordinator
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1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of settleable solids, total solids, total suspended solids, total dissolved solids, and suspended solids using gravimetric techniques. This SOP is based on the residue methods contained in *Methods for Chemical Analysis of Waters and Wastes* (MCAWW) and *Standard Methods for the Examination of Water and Wastewater* (SM).

1.2. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.3. This SOP is also applicable to solid and semisolid samples such as river and lake sediments, sludges separated from water and wastewater treatment processes and sludges from vacuum filtration, centrifugation, or other sludge dewatering processes following Standard Method 2540G.

1.4. The Total Solids (TS) protocol is based on MCAWW Method 160.3 and Standard Methods 2540B and 2540G.

1.5. The Total Dissolved Solids (TDS) protocol is based on MCAWW Method 160.1 and Standard Method 2540C.

1.6. The Total Suspended Solids (TSS) protocol is based on MCAWW Method 160.2 and Standard Method 2540D.

1.7. Methods 160.1, 160.2, 160.3 and 160.5 were withdrawn by the Methods Update Rule (MUR) of March 12, 2007, and replaced by the appropriate Standard Methods for the Examination of Water and Wastewater.

1.8. The method covers a range of 10 mg/L to 20,000 mg/L for TS and TDS. The range is 5 mg/L - 20,000 mg/L for TSS. As a practical matter, the final residue weight of a field sample shall be limited to between 2.5 mg and about 200 mg. For TS on solid samples, the range is 0.1% to 100% with an aliquot size of 5-10 g.

2. SUMMARY OF METHOD

2.1. Total Solids (TS): A well-mixed aliquot of the sample is quantitatively transferred to a pre-weighed evaporating dish and evaporated to dryness at 103-105°C. The increase in weight over that of the weight of the empty dish represents the total solids. For solid samples, the result is reported in % Total Solids.

2.2. Total Dissolved Solids (TDS): A well-mixed aliquot of the sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a pre-weighed evaporating dish, evaporated in an oven set slightly below boiling (98°C), and dried to

a constant weight at 180 ± 2°C. The increase in weight over that of the weight of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS determination.

2.3. Total Suspended Solids (TSS): A well-mixed aliquot of the sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to a constant weight at 103-105°C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination.

WARNING: Allow samples to cool before handling.

3. DEFINITIONS

3.1. Total Solids (TS): The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 103-105°C. Total solids includes "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

3.2. Total Dissolved Solids (TDS): Those solids passing through a 2.0 µm nominal pore size (or smaller) glass fiber filter and dried to constant weight at 180 ± 2°C. TDS is also referred to as filterable residue.

3.3. Total Suspended Solids (TSS): Those solids retained by a 2.0 µm nominal pore size (or smaller) glass fiber filter and dried to constant weight at 103-105°C. TSS is also referred to as non-filterable residue.

3.4. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.5. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. These materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2. Non-homogeneous samples may give erroneous results. Samples shall be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3. Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter shall be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts shall be noted in the Laboratory Information Management System (LIMS), refered to as TALS.

4.4. Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter. For samples with visible high TSS content, use a smaller volume.

4.5. Oil and grease in the samples may cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, shall not be included in the sample. The Project Manager must be informed and an anomaly filed when this type of sample is analyzed. Oil and grease can be included for TS analysis if dispersed with a blender prior to pouring.

4.6. Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7. The temperature at which the residue is dried has an important bearing on the results. Weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8. Pay close attention to desiccation after drying. Minimize opening the desiccator because moist air may enter. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

4.9. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

4.10. TDS samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.11. Total residue should be limited to about 200 mg as some samples dry with the formation of a crust that prevents water evaporation, requiring special handling to deal with this.

4.12. Some samples may have fine suspended solids, which pass through the glass fiber filter causing high TDS results.

4.13. Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.14. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1. Specific Safety Concerns or Requirements

5.1.1. The use of vacuum systems during sample filtering presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.2. Analysts must use tongs or heat protective gloves when handling hot materials being taken out of ovens.

5.1.3. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, PVC and nitrile gloves all provide adequate levels of protection against the chemicals used in this SOP.

5.1.4. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

There are no materials with a health rating of 3 or 4 used in this method.used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6. EQUIPMENT AND SUPPLIES

6.1. Analytical balance capable of weighing to 0.0001 g.

6.2. Vacuum filtration apparatus.

6.3. Vacuum pump equipped with moisture trap.

6.4. Glass fiber filter disks, 47 mm, without organic binder (eg. Whatman Filter Paper 934-AH) or equivalent, 2.0 µm nominal pore size (or smaller).

Note: Commercially available pre-weighed filters may be used for TSS analysis.

6.5. Desiccant containing a color indicator of moisture concentration or an instrumental indicator.

6.6. Drying ovens set at 103-105°C and 180 ± 2°C. Separate ovens should be maintained at appropriate temperatures if possible.

6.7. Thermometers, NIST traceable.

6.8. Conductivity meter and associated apparatus.

6.9. Glass beakers/evaporation dishes, 'to contain', assorted sizes.

6.10. Graduated cylinders, Class A, assorted sizes.

6.11. Volumetric flasks, Class A, assorted sizes.

6.12. Aluminum weighing dishes large enough to hold a 47 mm filter.

6.13. Forceps for handling filters.

6.14. Crucible tongs.

6.15. Zetex gloves or other gloves capable of providing protection at 180°C.

7. REAGENTS AND STANDARDS

7.1. Reagent water: Distilled or deionized water, free of analyte of interest. Water must contain particles no larger than 0.20 micron and have a resistance of at least 18 ohms.

7.2. LCS for TSS: Obtain a commercially prepared standard, such as ERA Hardness standard. True values vary depending on the specific lot obtained.

7.3. LCS for TDS and TS: Obtain a commercially prepared standard, such as ERA Minerals standard. True values vary depending on the specific lot obtained.

7.4. Spike solutions for matrix spikes: 5,000 mg/L NaCl. Weigh 5.0 g of reagent grade sodium chloride into a 1 L volumetric flask and dilute to volume with reagent water.

7.5. Commercially available reference materials may also be used.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in either plastic or glass bottles.

8.2. Samples must be stored at 4 ± 2°C to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

8.3. To achieve the reporting limits listed, the recommended minimum volumes are as follows:

Parameter	Volume (mL)	Reporting Limit
TS	100	10 mg/L
TDS	100	10 mg/L
TSS	100	5 mg/L

Smaller volumes can be used when high analyte levels are suspected. Reporting limits must be adjusted accordingly.

8.4. For TS on solid samples, the reporting limit is 0.1 % Total Solids, with an aliquot size of 5-10 g.

8.5. Low level TSS can be done with this method using 500 mL of sample. The reporting limit is 1 mg/L. The volume used for the prep blank must be the same as the sample. The LCS volume must be 100 mL to prevent filter overload. Low level TSS is done only if specifically requested by the client.

8.6. The holding times for TS, TDS, and TSS are 7 days.

8.7. The holding time for SS is 48 hours.

9. QUALITY CONTROL

The QC Program document (WS-PQA-003) provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.1. Table 1 at the end of this section provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.2. Initial Demonstration of Capability (IDOC): This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample containing the analyte(s) of interest that is used to monitor method

performance. The results of the initial demonstration study must be acceptable before the analyst performs the analysis of samples under this SOP. Further details are in Section 13.2.

9.3. Batch Definition: The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a MB, LCS, and a MS/MSD. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See policy WS-PQA-003 for further definition of the batch.

9.4. Method Blank (MB): One MB must be processed with each batch of 20 or fewer samples. The MB consists of reagent water that is carried through the entire analytical procedure, including filtration as applicable. The MB must be processed at the same time and in the same manner as the associated samples. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The volume of reagent water used must be the same as the volume listed in Section 8.3. Note: See 8.5 for low level TSS volume.

9.5. Laboratory Control Sample (LCS): One LCS must be processed with each batch of 20 or less samples. The LCS must be carried through the entire analytical procedure. The LCS must be processed at the same time and in the same manner as the associated samples. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Refer to Section 7 for instructions on how to obtain the LCS solutions. The LCS must be the same volume as the method blank, except for low level TSS. See Section 8.5.

9.6. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be processed with every process batch of similar matrix, not to exceed twenty (20) samples. The MS/MSD are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits for the LCS. Corrective actions must be documented in a Non-Conformance Memo (NCM), then implemented when recoveries of any spike analyte is outside control limits provided in LIMS or by the client. Reprocessing of the MB, LCS, the selected field sample(s), and/or the MS/MSD may be required after evaluation and review. Note: For TSS the MS/SD is not applicable. See Section 9.7 and 9.8.

9.7. A duplicate control sample (LCSD or DCS) may be substituted when insufficient sample volume is provided to process a MS/MSD pair if required by client or program. The LCSD is evaluated in the same manner as the LCS.

9.8. Sample Duplicate (DU): A duplicate sample is required for TSS analysis. Sample results should agree within 20 % if both the sample and sample duplicate results are >5X RL.

TABLE 1		
Acceptance Criteria and Corrective Actions-Residue		
QC Type	***Acceptance Criteria***	***Corrective Action***
Laboratory Control Sample (LCS)	TDS: 80%-120% Recovery TSS: 85%-115% Recovery TS: 80%-120% Recovery	Re-pour and reanalyze the batch unless the client agrees that another corrective action is acceptable.
Method Blank (MB)	±Reporting Limit	Re-pour and reanalyze all associated field samples with reportable levels of analyte. If all associated field sample results are ND, report the data and file an anomaly.
Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) Spike Level- (500 ppm)	TDS: 85%-115% Recovery, 20% RPD TS: 80%-120% Recovery, 20% RPD	Re-pour and reanalyze to confirm if RPD fails.
Sample Duplicate (DU)	20% RPD if sample and duplicate results are >5X the RL.	Re-pour and reanalyze for confirmation.
EC/TDS ratio	0.47 ~ 0.93	See Below

Table 2	
Corrective Actions for Residue	
Step 1: Investigate the physical properties of the sample.	1. Floatables, sediment content, viscosity, color, reaction to air exposure, smell, hygroscopic crystals, difficulty drying, etc. are enough to cause a failed EC/TDS ratio. Results can be reported with a note on the benchsheet when these apply. 2. If the sample spilled or splattered, or too much sediment was present, the sample must be reanalyzed.
Step 2: Reanalyze for EC	1. If the rerun EC confirms the original, and the EC/TDS ratio still fails, rerun TDS. 2. If the rerun EC differs from the original, and the EC/TDS ratio passes, report the TDS result and record new EC result. 3. EXCEPTIONS 3.1. If the EC is < 50 umhos/cm and TDS is < 200mg/L, report result. Note on the benchsheet that the EC/TDS ratio does not apply to samples with EC < 50 uhmos/cm. 3.2. If the EC is > 20,000 umhos/cm, and TDS is > 18,000mg/L, report result. Note on the benchsheet that the EC/TDS ratio does not apply to samples with EC > 20,000 uhmos/cm.
Step 3: Reanalyze for TDS	If the rerun TDS confirms the original, and the EC/TDS ratio still fails, report the original with a note on the benchsheet. If the rerun TDS differs from the original, and the EC/TDS ratio passes, report the rerun. File an HTV, if needed.

10. CALIBRATION

10.1. Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily, prior to sample analysis, by following the balance calibration SOP WS-QA-0041. Analytical balance calibration must be performed daily (every 24 hours).

10.1.1. Preventative Maintenance for Balances

On a daily basis, clean the pan and weighing compartment, to ensure that no dust or particles are interfering with the movement of the pan.

10.2. The oven temperature must be monitored using a calibrated thermometer on days the unit is in use and recorded in TALS and on the Oven Temperature Log, form QA-436, located on the oven. The Thermometer ID, oven Unit #, oven Location, and temperature range are recorded on the Oven Temperature Log form prior to use. Sample Status, Date, Observed/Corrected Temperature, Sample Batch number, and Initials are recorded on the Oven Temperature Log form when samples or sample remains are placed in the oven or taken out of the oven. If temperature readings fall outside of the acceptable range notify the QA Department and file a NCM. If the unit was used on a day the temperature was not recorded, contact the QA Department and the Department Manager. File a NCM for all affected samples.

10.2.1. Preventative Maintenance for Ovens

The electronics are serviced on an as-needed basis. The electronic read-outs are verified on an annual basis, or more frequently if checks against a NIST-traceable thermometer indicate a problem.

10.3. The conductivity of the water must be monitored and recorded daily in the Conductivity Logbook following the water monitoring SOP WS-QA-0014. The conductivity must be less than 1.0 umhos/cm (at 25°C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a NCM and must be approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified.

11.2. Any unauthorized deviation from this procedure must also be documented in a NCM with a cause and corrective action described.

11.3. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.4. Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filter holders must be done in a manner to ensure connection with the proper sample.

11.5. If possible, analyze all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.6. Note any sample abnormalities in TALS during pouring and after drying.

11.7. If there is limited sample volume or high solid content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted in TALS and reporting limits must be adjusted appropriately.

11.8. Proceed to the appropriate section for the desired method as follows:

Total Solids (TS)	11.9
Total Dissolved Solids (TDS)	11.10
Total Suspended Solids (TSS)	11.11

11.9. Total Solids, 2540B and 2540G

11.9.1. Preparation of Evaporating Dish

11.9.1.1. If only total solids are to be measured, heat clean dish to 103-105°C for one hour. Transfer dishes to dessicator for at least 1.5 hours.

Note: To maintain consistent conditions, the drying time and cooling time between weighings should be the same.

11.9.1.2. Weigh immediately to the nearest 0.1 mg. Record the weight in TALS.

11.9.1.3. Store the dish in the desiccator until ready to use.

11.9.2. Sample Preparation

Prescreen the samples using a calibrated conductivity meter to determine the required sample volume or dilution. Record the sample conductivity in TALS. See Section 12.1 for formula, if needed.

11.9.3. Sample Analysis

11.9.3.1. Transfer a measured aliquot of well-mixed sample to the pre-weighed, labeled dish. Record the volume of the sample to the nearest mL in TALS.

11.9.3.2. Record the date and time the samples were poured. This will be used as the analysis time.

11.9.3.3. For soil and solid samples, weight 5-10 grams of well-homogenized sample into the dish.

11.9.3.4. For the LCS, measure 100 mL of the LCS solution (Section 7.2) and pour into the dish.

11.9.3.5. For the MB, measure 100 mL of reagent water and pour into the dish.

11.9.3.6. For MS/MSD aliquots, add 10 mL of spike solution (Section 7.3) to the sample in the dish.

11.9.3.7. Evaporate the sample to dryness in a drying oven. The temperature should be lowered down to 98°C to prevent boiling and splattering of the sample.

11.9.3.8. Dry the evaporated sample for at least one hour at 103-105°C. Dry soil and solid samples for % TS for a minimum of 12 hours at 103-105°C.

11.9.3.9. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven and place in a desiccator and cool to room temperature for 1.5 hr.

11.9.3.10. Weigh the dish to the nearest 0.1 mg. Record the weight in TALS.

11.9.3.11. Return the samples to the oven for another hour, then cool in a desiccator for 1.5 hours, and reweigh. If the weights are constant, analysis is complete. If the weights vary by more than 4% of the previous weight or 0.5 mg (whichever is less), repeat the drying, cooling, desiccating and weighing process.

Note: For residue values > 12.5 mg, 0.5mg is an appropriate cut-off for determining constant weight.

Note: When weighing a dried sample, be alert to changes in weight due to air exposure and/or sample degradation. Minimize time spent in weighing or dish exposure to ambient air.

11.9.3.12. If sample fails to reach a constant weight after three weighings, the lowest final weight is used and an NCM is filed.

11.9.3.13. See Sections 12.2 and 12.3 for calculation equations, if needed.

11.10. Total Dissolved Solids, 2540C

11.10.1. Preparation of Evaporating Dishes

11.10.1.1. If only total dissolved solids are to be measured, heat clean dish to 180 ± 2°C for one hour. Transfer dishes to dessicator for at least 1.5 hours.

11.10.1.2. Weigh immediately to the nearest 0.1 mg. Record the weight in TALS.

11.10.1.3. Store in the desiccator until ready to use.

11.10.2. Sample Preparation

Prescreen the samples using a calibrated conductivity meter to determine the required sample volume or dilution. See Section 12.1 for formula, if needed.

Note: TDS is typically 55%-90% of the conductance result. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations, for samples containing non-ionic species, or for samples with conductance greater than 10,000 umhos/cm or less than 10 umhos/cm. 2 mL is the smallest volume that can be used for analysis.

Conductance Value (umhos/cm)	Sample Volume (mL)
<2,500	100
2,500-5,000	50
5,000-7,500	25
7,500-10,000	10
10,000-20,000	5
>20,000	2

11.10.3. Sample Analysis

11.10.3.1. Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample.

11.10.3.2. Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: If the sample also requires TSS, pre-weigh the prepared filter and refer to Section 11.11 for additional guidance.

11.10.3.3. Shake the sample vigorously and rapidly transfer 100 mL (or a smaller portion which will yield between 10 and 200 mg dried residue) to the filter funnel by means of a graduated cylinder. If more than 10 minutes are required to complete filtration, decrease sample size.

Note: For samples high in sediment that need to be analyzed for TDS only (meaning that the sample bottle will not be used for any other analyses), do not shake the samples prior to analysis. TDS is a measure of the salts dissolved in the aqueous sample. Additionally, multiple filters may be used if performing only TDS analysis (and not TSS).

11.10.3.4. For the MB, process 100 mL of reagent water.

11.10.3.5. For the LCS, process 100 mL of the LCS solution. Refer to Section 7.3 for instructions on how to obtain the LCS.

11.10.3.6. For MS/MSD aliquots, add 10 mL of the spiking solution (Section 7.3).

11.10.3.7. Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow for complete drainage between washings. Continue to apply vacuum after filtration is complete to remove as much water as possible.

11.10.3.8. Transfer the filtrate (including the washings) to a pre-weighed evaporating dish. Rinse the receiving flask with 10-25 mL of

reagent water and transfer washings into the dish to ensure complete transfer of the sample.

Note: Record the date and time the samples were poured. This will be used as the analysis time.

11.10.3.9. Evaporate the samples to dryness in an oven set slightly below boiling (98°C). If filtrate volume exceeds dish capacity, add successive portions to the same dish after evaporation.

11.10.3.10. Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

11.10.3.11. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven and place in a desiccator and cool to room temperature for 1.5 hr.

11.10.3.12. Immediately weigh the dish to the nearest 0.1 mg. Record the weight in TALS.

11.10.3.13. Return the samples to the oven for another hour, cool in a desiccator for 1-1.5 hours, and reweigh. If weights are constant, analysis is complete. If weights vary by more than 4% of the previous weight or 0.5 mg (whichever is less), repeat the drying, cooling and desiccating process.

Note: For residue values > 12.5 mg, 0.5mg is an appropriate cut-off for determining constant weight.

11.10.3.14. If sample fails to reach a constant weight after three weighings, the lowest final weight is used and an NCM is filed.

11.10.3.15. See Sections 12.4 and 12.5 for calculation equations, if needed.

11.11. Total Suspended Solids, 2540D

11.11.1. Preparation of Glass Fiber Filter Disc

Note: As an alternative to the steps outlined below, prepared and pre-weighed filters may be purchased for use under this method.

11.11.1.1. Place the glass fiber filter discs, one at a time, in the membrane filtration apparatus.

11.11.1.2. While the vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water.

11.11.1.3. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.

11.11.1.4. Remove the filter from the membrane filtration apparatus, place in a labeled aluminum weighing dish and dry in an oven at 103-105°C for one hour.

11.11.1.5. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven, place in a desiccator and cool to room temperature for one hour.

11.11.1.6. Weigh the cooled filter using an analytical balance to the nearest 0.1 mg. Record the weight in TALS. Handle the filters only with forceps.

11.11.2. Selection of Sample Volume

11.11.2.1. For a 47 mm diameter filter, filter 100 mL of sample or less depending on the visible particulates present in the sample. Limit the sample size to that yielding no more than 200 mg residue.

11.11.2.2. If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

11.11.2.3. Record the sample volume used in TALS.

11.11.3. Sample Analysis

11.11.3.1. Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water and begin suction.

Note: Handle the filters only with forceps.

11.11.3.2. Shake the sample vigorously and quantitatively transfer 100 mL (or an appropriate aliquot) of the sample to the filter using a graduated cylinder or pipet.

Note: If Total Dissolved Solids (TDS) is also required, the filtrate may be used. Refer to Section 11.10 for additional guidance.

11.11.3.3. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

11.11.3.4. With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

11.11.3.5. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

11.11.3.6. Carefully remove the filter from the filter support and transfer to a weighing dish or aluminum pan.

11.11.3.7. Record the date and time the samples were poured. This will be used as the analysis time.

11.11.3.8. Dry the filter for at least one hour at 103-105°C.

11.11.3.9. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven and place in a desiccator and cool to room temperature for one hour.

11.11.3.10. Immediately weigh the filters to the nearest 0.1 mg. Record the weight in TALS.

11.11.3.11. Return the samples to the oven for another hour, cool in a desiccator for 1 hour, and reweigh. If weights are constant, analysis is complete. If weights vary by more than 4% of the previous weight or 0.5 mg (whichever is less), repeat the drying, cooling and desiccating process.

11.11.3.12. If sample fails to reach a constant weight after three weighings, the lowest final weight is used and an NCM is filed.

11.11.3.13. See Section 12.6 for calculation equations, if needed.

12. CALCULATIONS/DATA REDUCTION

12.1. The following formula should be used to approximate the required sample volume:

(2000 x 100)/Conductivity = mL sample to use

Note: To avoid "oddball" reporting limits, round the determined sample volume to one of the following volume increments: 2, 5, 10, 20, 25, 50 or 100 mL.

12.2. Calculate **Total Solids, 2540B** as follows:

$$\text{Total Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg) (Use lowest final weight achieved.)
B = weight of dish (mg)
C = volume of sample (mL)

12.3. Calculate **% Total Solids, 2540G** as follows:

% Total Solids = ((A-B) x 1000)/(C-B)

Where: A = weight of dried residue + dish (mg) *(Use lowest final weight achieved.)*
B = weight of dish (mg)

C = weight of wet sample + dish (mg)

12.4. Calculate **Total Dissolved Solids, 2540C** as follows:

$$\text{Total Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg) (Use lowest final weight achieved.)
B = weight of dish (mg)
C = volume of sample (mL)

12.5. Calculate the expected TDS range from conductance (as umhos/cm).

Expected TDS_{low} (mg/L) = (Conductance x 0.55)

Expected TDS_{high} (mg/L) = (Conductance x 0.9)

12.6. Calculate **Total Suspended Solids, 2540D** as follows:

$$\text{Total Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of filter + residue (mg) (Use lowest final weight achieved.)
B = weight of filter (mg)
C = volume of sample filtered (mL)

12.7. The lowest final sample weight is used for calculating solids content.

12.8. If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.2.1. MDLs and MDL verifications for TS and TDS are conducted using the NaCl spike described in Section 7.4 diluted as needed.

13.2.2. MDLs and MDL verifications for TSS are conducted using the LCS solutions (Section 7.2). The appropriate volume to provide approximately 5mg/L in 100 mL is determined, and that volume is added to sufficient reagent water to

make a total of 100 mL in the funnel.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. For TS and TDS, four aliquots are prepared. Each aliquot consists of 0.8 mL of the NaCl matrix spike solution (Section 7.4) and 100 mL reagent water. These four aliquots analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of each aliquot is 40 (39.6825) mg/L.

13.3.2. For TSS, four aliquots are prepared using the LCS solution (Section 7.2). These four aliquots analyzed using the same procedures used to analyze samples, including sample preparation.

13.3.3. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.5. The reporting limit is 10 mg/L for TS, and TDS.

13.6. The reporting limit is 5 mg/L for TSS.

13.7. The Initial Demonstration of Capability study as detailed in Section 9.2 must be acceptable before the analysis of field samples under this SOP may begin.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

15.1. Used filters, miscellaneous disposable glassware and contaminated gloves are disposed of to the hazardous lab trash buckets. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Liquid samples that have been filtered as part of the analytical process are collected in a satellite carboy. When full or after no more than one year, this carboy is moved to the waste disposal area for consolidation and disposal.

16. REFERENCES/CROSS REFERENCES

16.1. Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Methods 160.1, 160.2, 160.3, 160.4 and 160.5.

16.2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992: Methods 2540A-G and 2510A.

16.3. Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995: Methods 2540A-G and 2510A.

16.4. Standard Methods for the Examination of Water and Wastewater, Methods 2540B-1997, 2540C-1997, 2540D-1997 and 2510B-1997..

16.5. 40 CFR Parts 122 and 136.

16.6. Quality Control Program, WS-PQA-003, current revision.

16.7. Calibration and Calibration Check of Balances, WS-QA-0041, current revision.

16.8. Monitoring of Reagent-Grade Laboratory Water, WS-QA-0014, current revision.

17. METHOD MODIFICATIONS

17.1. Samples are not taken through the drying, cooling, and weighing cycle more than three times. If the sample fails to reach constant weight within three cycles the lowest final weight is used for the calculation, and an NCM is filed.

17.2. The filter cleaning procedure outlined in Method 160.1 and SM2540C is not applied. This SOP requires the use of method blanks to document system cleanliness.

17.3. All samples are not analyzed in duplicate. Instead, one sample duplicate or a matrix spike and matrix spike duplicate are analyzed per batch. This allows for precision monitoring.

17.4. Additional sample is not added to the dish until the residue is at least 25 mg. Instead, the volumes listed in Section 8.3 are used to obtain the established reporting limits. Dilutions can be made when high analyte levels are suspected.

17.5. Duplicates must agree within 20% of each other, instead of 5% of their average, as listed in reference method 2540.

17.6. Method 160.3 (TDS), Section 7.5 specifies that samples are evaporated to dryness on a steam bath. This SOP specifies (Section 11.10.3.8) that samples are evaporated to dryness in an oven set slightly below boiling . Both techniques accomplish the same goal, to reduce spattering and potential loss of sample during evaporation.

17.7. SM 2540D specifies transferring a stirring sample using a pipette. This SOP specifies (Section 11.11.3.2) that a vigorously shaken sample aliquot may be quantitatively transferred by graduated cylinder or pipette.

17.8. SM 2540D indicates that the washed and dried filter should go through more than one weighing cycle to establish a constant weight. This SOP specifies (Section 11.11.1) that the filter is weighed only once.

18. ATTACHMENTS

18.1. No attachments are present.

19. REVISION HISTORY

19.1. WS-WC-0002 Revision 4.3, Effective 11/22/2013

19.1.1. Removed all references to MCAWW 160.4 (Title,

19.1.2. Removed all references to SM2540F (Settleable Solids) as the method is not currently performed at the facility. (Title, Scope (1.4), Summary of Method (2.4), Definitions (3.3), Sampling (8.3), Procedure (11.12)

19.1.3. Revised the procedures to reflect use of the LIMs rather than a benchsheet for recording data.

19.1.4. Updated Section 13 to include details for MDLs and IDOCs.

19.1.5. Corrected revision history for revisions 4.1 and 4.2.

19.1.6. Editorial revisions

19.2. WS-WC-0002, Revision 4.2, Effective 8/3/2012

19.2.1. Table 1: Inserted number value (.47 – .93) in place of percentages.

19.2.2. Section(s) 11.9.1.1 and 11.10.1.1 – added “Transfer dishes to dessicator for at least 1.5 hours.

19.2.3. Sections 11.9.3.10, 11.10.3.12, and 11.11.3.11 – Deleted “If weights still vary by more than 0.5 mg, file an NCM and report the data.”

19.2.4. Editorial revisions.

19.3. WS-WC-0002, Revision 4.1, Effective 04/17/09

19.3.1. Removed all references to solid volatiles, solid dissolved volatiles, solid suspended volatiles, and muffle furnaces.

19.3.2. Updated SOP references to correct versions.

Title: Determination of Anions by Ion Chromatography [EPA Method 300.0-Part A, SW-846/Method 9056]

Approvals (Signature/Date):

Thep Phomsopha Technical Manager	7/15/14 Date	Joe Schairer Health & Safety Manager / Coordinator	7/16/14 Date
Lisa Stafford Interim Quality Assurance Manager	7/17/2014 Date	Robert Hrabak Laboratory Manager	7/16/14 Date

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures for analysis of inorganic anions by Ion Chromatography (IC) using EPA 600/Method 300.0 Part A and SW-846/Method 9056 as method references.

1.2. Applicable Matrices:

1.2.1. Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, and reagent water.

1.2.2. Solutions collected from bomb combustion extraction of solid waste samples by SW-846/Method 5050.

1.2.3. Solids after extraction by deionized water leaching procedure as outlined in SOP WS-WC-0049 (based on Method 300.0, Section 11.7).

1.2.4. Other leachates (when no acetic acid is used).

1.3. This method covers the determination of the following inorganic anions as well as the retention times (recommended, may vary depending on the eluent strength), individual reporting limits, and analytical linear ranges:

Anions	**Approximate Retention time (min.) with ±% window size**		**Water Reporting Limit (mg/L)**	**Soil Reporting Limit (mg/kg)**	**Analytical Linear Range (mg/L)**
Fluoride, (F^-)	2.0	5%	0.5	2.5	0.5 - 50
Chloride, (Cl^-)	2.9	5%	1.0	5.0	1 - 100
Nitrite, (as N)	3.6	5%	0.05	0.25	0.05 - 10
Bromide, (Br^-)	4.3	5%	0.5	2.5	0.5 - 50
Nitrate, (as N)	5.0	5%	0.05	0.25	0.05 - 10
Ortho-Phosphate (as P)	6.2	5%	0.20	1.0	0.20 - 20
Sulfate, (SO_4)	7.1	5%	1.0	5.0	1 - 100

1.4. The analytical time per injection is approximately 10 minutes from injection to chromatogram printout.

1.5. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

1.6. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. A small volume of sample, typically 2 to 5 mL, is injected into an ion chromatograph to flush and fill a constant volume in the sample loop. The sample is then injected into a stream of carbonate-bicarbonate eluent and pumped through two different ion exchange columns, a suppressor device and into a conductivity detector.

2.2. Two columns, the guard column and a separator column, are packed with a low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The suppressor device reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell.

2.3. Anions are identified based on their retention times and quantified using the peak area in comparison to the calibration curve generated from known standards.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention time.

4.2. Samples that contain particulates larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems. Particulates can be separated by filtering the samples, standards, or reagents through a filter syringe with a 0.45 micron filter cartridge.

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in ion chromatographs.

4.4. Low molecular weight organic acids, such as formate, acetate, propionate, etc… have retention times similar to fluoride and can make it difficult to quantify fluoride content of the sample.

4.5. The water dip or negative peak that elutes near the front end can interfere with the fluoride peak. It can be eliminated by the addition of the equivalent of 1 mL of concentrated eluent to 100 mL of each standard and sample.

4.6. Any anion that is not retained in the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant.

4.7. The acetate anion elutes early during the chromatographic run. The retention times of target analytes shift when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

4.8. Nitrite may be oxidized to form nitrate by bacteria in water solutions. It is recommended that standard solutions of nitrite and nitrate be stored separately until just prior to analysis.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), theSacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Sodium Fluoride is Highly Toxic.

5.1.2. Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.3. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. PVC, latex and nitrile gloves provide adequate levels of protection against the chemicals used in this SOP.

5.1.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Reagent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials Section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Potassium Bromide		None Established	Inhalation of dust may cause irritation to respiratory tract, with coughing. Dust may irritate skin, with redness and itching. Solutions may cause redness and burns.
Sodium Fluoride	Poison	2.5 Mg/M^3-TWA as F	**Highly Toxic.** Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.
Sodium Nitrite	Oxidizer Poison Reactive	None	**Danger! Strong oxidizer and toxic material. Contact with other material may cause fire. Heat, shock, or contact with other material may cause fire or explosive decomposition.** Causes irritation to the respiratory tract and systemic poisoning. Can irritate the mouth, esophagus, stomach, etc. Excessive amounts affect the blood and blood vessels. Signs and symptoms of nitrite poisoning include intense cyanosis, nausea, dizziness, vomiting, collapse, spasms of abdominal pain, rapid heart beat, irregular breathing, coma, convulsions, and death due to circulatory collapse. Causes irritation, redness and pain to the skin and eyes. May be absorbed through the skin causing systemic poisoning.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Ion chromatograph - This method uses an IC instrument manufactured by Dionex, Model # ICS-1000 utilizing a Windows® based Chromeleon® Chromatography

Workstation (Rev. 6.5) for instrument control, digital data collection and processing software. It is equipped with an injection valve, auto sampler, pump with a flow rate of 1.2 mL/min., integrator, a 10-uL loop, and set up with the following components:

6.1.1. Anion guard column: Placed before the separator column to protect the separator column from being fouled by particulates or certain organic constituents, (Ion Pac AG22A, 4mm, Dionex P/N 072784).

6.1.2. Anion separator column: Packed with low-capacity pellicular anion exchange resin that is styrene divinylbenzene-based, (Ion Pac AS22A, 4mm, Dionex P/N 072782).

6.1.3. Anion Self Regenerating Suppressor: Capable of converting the eluent and separated anions to their respective acid forms, (ASRS 300, 4mm, Dionex P/N 04554).

6.1.4. Detector: Flow through conductivity cell measures electrical conductance of analyte ions, (D56, Dionex P/N 057985).

6.2. Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.3. Syringe, disposable, 2-10 mL capacity and equipped with male pressure fitting.

6.4. 0.45 micron acrodisk filter.

6.5. IC sample vial and filter caps - at least 5 mL capacity

6.6. Test strips for Nitrite, Nitrate, Chloride, and Sulfate.

6.7. Various analytical glassware of different sizes - graduated cylinder, volumetric flask, pipettes, etc.

6.8. Preventative and routine maintenance as described in the “Schedule of Routine Maintenance” in the QAM.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2. Reagent Water: Distilled or deionized water free of the anions of interest. Water shall contain particles no larger than 0.20 micron, have maximum conductivity of 1µmhos/cm, and have a maximum resistivity of at least 1 Mohm/cm at 25°C (per SOP WS-QA-0014).

7.3. A commercially available stock eluent, such as the AS22 Eluent Concentrate (P/N 0963965) – 0.45 M Na_2CO_3 / 0.14 M $NaHCO_3$. from Dionex may be purchased. Follow manufacturer's instructions to obtain 4.5 mM Sodium Carbonate/1.4 mM Sodium Bicarbonate eluent working solution.

7.3.1. As an alternative, Eluent Stock Solution: Dissolve 47.7 g Na_2CO_3 and 11.76 g $NaHCO_3$ in approximately 500 mL reagent water in a 1000 mL flask. Bring to a final volume of 1000 mL with reagent water. This solution is good for one year.

7.3.2. Eluent Working Solution: Add 20 mL of the eluent stock to approximately 1000 mL of reagent water in a 2000 mL volumetric flask. Bring to a final volume of 2000 mL. This solution expires 6 months from the date of preparation.

7.4. Stock Mixed Anion Calibration Standards: Commercially available, custom mixed anion standards, such as AccuStandard IS-14976-R3. These include AccuStandard custom IC mixes IS-14936 and IS-14937. This standard contains 500µg/L Fluoride, 500 µg/L Chloride, 500 µg/L Bromide, 100 µg/L Nitrate (as N), 200 µg/L Phosphate (as P), and 500 µg/L Sulfate, at certified concentrations. This is stored at room temperature, and expires one year from the open date, or the vendor expiration date, whichever is lesser. The current calibration curve values do not allow for calibration curve preparation from a single stock Mixed Anion Calibration Standard solution.

7.5. Stock Single Analyte Calibration Standards, 1000 mg/L (1 mg/mL): Stock standards are purchased as certified solutions. Stock standard solutions are stored at room temperature and expire one year from the open date, or the vendor expiration date, whichever is lesser.

7.6. Stock Mixed Anion Reference Standard: Commercially available, ready-to-use custom mixed anion standard, such as AccuStandard custom IC mix IS-16268-R2. This standard is from a different source than the Stock Mixed Anion Standard Calibration Solutions. This standard contains 15 µg/L Fluoride, 15 µg/L Chloride, 15 µg/L Bromide, 3.15 µg/L Nitrate (as N), 6 µg/L Phosphate (as P), and 15 µg/L Sulfate, at certified concentrations. This is stored at room temperature, and expires one year from the open date, or the vendor expiration date, whichever is lesser.

7.7. Stock Single Analyte Reference Standards, 1000 mg/L (1 mg/mL) are also purchased as certified solutions. These standards are from a different source than the Stock Single Analyte Calibration Standards. Stock Standard solutons are stored at room

temperature and expire on year from the open date, or the vendor expiration date, whichever is lesser.

7.8. Stability of Stock Standard Solutions: All purchased certified stock solutions are good for one year of opening, or manufacturer expiration date, whichever is lesser.

7.9. Anion Working Calibration and Reference Standards: A reagent blank consisting of reagent water, and a series of standards with different levels of concentrations of all anions combined are prepared from the Stock Mixed Anion Calibration and Reference Standards if applicable (Sections 7.4 and 7.6, respectively). Alternatively, the standards may be prepared from Stock single analyte Calibration and Reference Standards (Sections 7.5 and 7.7, respectively). These combination anion solutions are used to calibrate the instrument monthly or when columns or eluent strengths are changed, or when new eluent stock solution is made. Working standards are to be stored at 4 ± 2°C.

7.10. See the following tables for Standard Curve Levels and Preparation recipes.

Table 1. Calibration and Reference Concentration Levels, mg/L

ANIONS	ICV[a]	Level 0[b]	Level 1	Level 2	Level 3[c]	Level 4
		Autocal 0	Autocal 1R	Autocal 2R	Autocal 3R	Autocal 4R
Fluoride (F^-)	7.5	0.0	0.5	5	10	20
Chloride (Cl^-)	7.5	0.0	1.0	5	10	20
Nitrite (NO_2^- as N)	3.75	0.0	0.05	0.5	5	10
Bromide (Br^{--})	7.5	0.0	0.5	5	10	20
Nitrate (NO_3^- as N)	1.875	0.0	0.05	0.5	5	10
Ortho-Phosphate (as P)	3	0.0	0.2	1	5	10
Sulfate (SO_4^{2-})	7.5	0.0	1.0	5	15	50

[a] Prepared from Stock Mixed Anion Reference Standard and Nitrite Stock Single Analyte Reference Standard. Alternately prepared from Stock Single Analyte Reference Standards.
[b] Level 0 is reagent water
[c] Mid-level standard for continuing calibration verification (CCV)

Table 2. Standard Curve Preparation Recipe (mL[a])

Anions	ICV[b]	ICV[c] Alt	ICV[d] Alt2	Level 1 AutoCal 1R	Level 2 AutoCal 2R	Level 3[e] AutoCal 3R	Level 3[f] Alt	Level 4 AutoCal 4R
Final Volume	100	100	5	500	100	250	100	100
Fluoride (F^-)	0.75[b]	NA	NA	0.25	0.5	2.5	1	2
Chloride (Cl^-)	0.75[b]	NA	NA	0.5	0.5	2.5	1	2
Nitrite (NO_2 as N)	0.375[b]	NA	NA	0.025	0.05	1.25	0.5	1
Bromide (Br^-)	0.75[b]	NA	NA	0.25	0.5	2.5	1	2
Nitrate (NO_3^- as N)	0.1875[b]	NA	NA	0.025	0.05	1.25	1	1
Ortho Phosphate (as P)	0.30[b]	NA	NA	0.1	0.1	1.25	0.5	1
Sulfate (SO_4^{2-})	0.75[b]	NA	NA	0.5	0.5	6.25	2.5	5
Stock Mixed Anion Reference Standard	NA	50	1.5	NA	NA	NA	NA	NA
Nitrite (NO_2 as N)[g]	NA	0.375	0.01875	NA	NA	NA	NA	NA

[a] Prepared from 1000 µg/L Stock Single Analyte Calibration Standards except for ICV, ICV Alt and ICV Alt 2.

[b] ICV prepared from 1000 µg/L Stock single Analyte Reference Standards .

[c] ICV Alt prepared from Stock Mixed Anion Reference Standard and Nitrite 100 µg/L Stock Single Analyte Reference Standard.

[d] ICV Alt2 prepared daily as needed from Stock Mixed Anion Reference Standard and Nitrite 1000 µg/L Stock Single Analyte Reference Standard. This meets CWA and NPDESAS requirements.

[e] Mid-level standard for Continuing Calibration Curve (CCV).

[f] Level 3 Alt is prepared daily as needed. Level 3 Alt can be prepared monthly from all anions **EXCEPT** Nitrite, which must be added daily. In this case 0.025mL of 1000µg/L Nitrite Stock Single Analyte Calibration Standard is added to a 5mL aliquot of the Level 3 Alt solution to generate the daily working standard/CCV. This meets CWA and NPDES requirements.

[g] This solution must be a 1000 µg/L Stock Single Analyte Reference Standard.

7.11. Preparation of Calibration Standard Curve Solutions:

7.11.1. Level 0- Reagent water.

7.11.2. Level 1R- 4R and ICV solutions are prepared using the standards and volumes indicated in Table 2.

7.11.3. Working Reference Solution: A second-source reference solution is prepared as needed. This solution is analyzed daily as the Initial Calibration Verification (ICV). The second source Working Reference Solution (ICV) is at or near the mid-point of the calibration range.

7.12. Stability of Anions Working Calibration and Reference Standards: Based on the laboratory's stability study, all of the combined solutions in Tables 1 and 2 are stable for one month. Working Calibration Reference Standards must be prepared fresh daily (specifically the Nitrite component) when samples are being processed under the Clean Water Act (CWA) or NPDES programs. This includes the CCV and ICV.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in pre-cleaned plastic and glass bottles. The volume collected should be sufficient to ensure a representative sample, allow replicate analysis when needed and minimize sample disposal.

8.2. Preservation, holding time and minimum sample requirements are listed in the following table:

TABLE 3				
Preservation, Holding Time, and Minimum Sample Size				
Analyte	**Preservation**	**Water Holding Time[(e)]**	**Minimum Sample Size**	
			Water	**Soil**
Fluoride (F^-)	4ºC	28 days	50 mL	50 g
Chloride (Cl^-)	4ºC	28 days	50 mL	50 g
Nitrite (as N)	4ºC	48 hours	50 mL	50 g
Bromide (Br^-)	4ºC	28 days	50 mL	50 g
Nitrate (as N)	4ºC	48 hours	50 mL	50 g
Ortho-Phosphate (as P)	4ºC	48 hours	50 mL	50 g
Sulfate (SO_4)	4ºC	28 days	50 mL	50 g

[(e)] Holding times are measured from date of sampling.

8.3. Holding times for soils are measured from the date/time of the generation of the DI water leachate. The holding time of the leachate follows that of the water samples holding time.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability - See Section 13.

9.2. Batch definition-The batch is a set of up to 20 samples of the same matrix processed using the sample procedures and reagents within the same time period. The Quality Control Batch must contain a Matrix Spike/Matrix Spike Duplicate (MS/MSD), a Laboratory Control Sample (LCS) and a Method Blank (MB). For Method 9056, or in some cases at client request, the MS/MSD may be replaced with a Matrix Spike and Sample Duplicate (MS/DU). Method 9056 also requires the analysis of a sample duplicate for one in every ten samples.

9.2.1. Batches for all water samples are defined at the analytical level and are specific to individual instruments. Refer to the QC Program document (PQA-003) for further details of the batch definition. Batches for all soil/solid samples are defined at the sample preparation stage. Prep batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. See SOP WS-WC-0049 for additional information on the soil/solid preparation procedure.

9.2.2. Standards/samples are evaluated separately for each individual anion. Example: If an LCS fails for one anion, all associated samples being analyzed for that particular anion are subject to corrective actions. All other anions can be reported with no corrective action, assuming that all other QC criteria has been met for the batch. See Table A for acceptance criteria and corrective actions.

9.2.3. Method Blanks (MB)-One method blank must be prepared with every process batch of similar matrix, not to exceed twenty samples. The method blank is carried through the entire analytical procedure and must have a concentration of less than the reporting limit for the analyte of interest, or less than ½ the reporting limit for projects covered by the DoD QSM.

9.2.3.1. ICB-analytical blank made of reagent water, analyzed after the ICV and evaluated as the MB.

9.2.3.2. CCB-analytical blank made of reagent water, analyzed after each CCV.

9.2.3.3. Soil/solid samples-a water blank taken through the preparation procedure is analyzed during the run in addition to the ICB/CCBs and evaluated as the MB.

9.2.4. Laboratory Control Sample (LCS)-An LCS must be analyzed with every process batch of similar matrix, not to exceed twenty samples. The LCS contains known levels of the analytes of interest.

9.2.4.1. ICV/REF/LCS-daily initial calibration verification from a second source analyzed to verify instrument calibration and evaluated as the LCS. Acceptance criteria: 90%-110%.

9.2.4.2. CCV-a midlevel standard, preferably the 3R calibration standard, analyzed after every ten samples and at the close of the analytical run. The concentration of the CCV standard shall be between the low calibration standard and the mid-point of the calibration range. For Method 300.0 compliance, each CCV must be within 90%-110% (± 10%) of the true value. If a CCV fails the criteria, all

samples surrounding the failed CCV should be reanalyzed for the out of control anions. For Method 9056 compliance, in addition to evaluating the drift from the calibration (i.e., ±10% of the true value), evaluate the overall instrument drift. Each CCV must be within ± 5% of the response of the preceding CCV, in addition to ± 10% of the true value. If one of the criteria is not met for a particular anion, all samples surrounding the failed CCV should be reanalyzed for the out of control anion. For more information, refer to WS-PQA-003.

9.2.4.3. The LCS for soil/solid samples is prepared from a water blank taken through the preparation procedure. The LCS is spiked after the prep and prior to the analysis. Acceptance criteria: 85%-115%.

9.2.5. Matrix Spikes (MS/MSD)-A Matrix Spike/Matrix Spike Duplicate pair must be analyzed with every process batch of similar matrix, not to exceed twenty samples. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration where the spike level should be at or near the concentration used for the LC, or the mid-point of the curve. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Acceptance criteria for aqueous samples is 90% - 110%. For solids and wipes the acceptance criteria is 85%-115%, 20% RPD.

9.2.5.1. Additionally the MS/MSD for soil/solid samples is prepared from a sample duplicate taken through the preparation procedure. The MS/MSD is spiked after the prep and prior to the analysis.

9.2.6. Laboratory Control Sample Duplicate (LCSD/LCSD)-A duplicate control sample may be substituted when insufficient sample volume is provided to process an MS/MSD pair as required by program or client. The LCSD is prepared, analyzed and evaluated in the same manner as the LCS.

9.2.6.1. An additional ICV/REF/LCS standard can be analyzed during the run, or two successive CCVs can be reported as the LCS and LCSD.

9.2.6.2. Soil/solid samples - an additional aliquot of the LCS water blank is taken, prepared and analyzed in the same manner as the LCS.

9.2.7. See Table A for acceptance criteria and corrective action.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

10.2. Prepare standards containing each analyte of interest at a minimum of 4 calibration levels and a reagent blank. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Refer to 7.10 and 7.11 for standard levels and preparation.

10.3. Initial Calibration (Demonstration of Linear Calibration Range)

10.3.1. A new calibration curve must be generated monthly or sooner if:

Major changes are made to the system or when the daily continuing calibration verification (ICV/CCV) criteria cannot be met. Major changes include new columns, or any changes in instrument operating parameters, including eluent flows, eluent strength, eluent stock solutions, etc.

10.3.2. The initial calibration curve must be verified daily by the analysis of a mid-level continuing calibration standard (ICV).

10.3.3. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach introduce each calibration standard into the IC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

10.3.4. The curve utilizes a linear regression where the correlation coefficient of each analyte, r, must be $\geq$ 0.995 for the initial calibration to be acceptable.

NOTE: Discrepancies may exist between the ICAL: as evaluated using instrument software and the ICAL as evaluated using Chrom, resulting in the continued use of a failing ICAL.

The ICAL data in Chrom is to be evaluated as soon as possible and prior to starting a new analytical sequence to prevent the use of a failing ICAL.

10.4. Retention Times

10.4.1. The instrument records retention times for each anion during the initial calibration.

10.4.2. Retention time of target analytes shift as a function of total anion concentration. Nitrate and sulfate exhibit the greatest retention shifts, although all anions are affected to some degree. In some cases, this peak migration may produce poor resolution or identification.

10.4.3. The width of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards over

the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.4.3.1. Retention time window study shall be performed after major maintenance which affects the chromatography system (e.g. column change).

10.4.4. The approximate retention time and % window size is listed in Section 1.3, which is based on historical performance.

10.4.4.1. Criteria: The maximum allowable shift in the retention time/ instrument response is 10%.

10.4.4.2. Corrective Action: Repeat analysis using fresh calibration standards. If the results are still more than 10%, a new calibration curve must be prepared for that analyte.

10.5. Nonlinear response can result when the separator column capacity is exceeded (overloading). Maximum column loading (all anions) should not exceed about 400 ppm.

10.6. Initial and Continuing Calibration Verification (ICV/CCVs and ICB/CCBs):

10.6.1. ICV/REF - The daily calibration verification must be analyzed at the start of each run, and once every 24 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new ICV run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with an ICV.

10.6.2. CCV - analyzed every 10 injections and at the end of the analytical sequence. Evaluated in the same manner as the ICV.

10.6.3. ICB/CCB - Reagent water that is immediately analyzed following the ICV/CCV analysis.

10.6.4. Details of ICV/ICB and CCV/CCB acceptance are detailed in Table A.

10.7. Examples of analytical sequences (including frequency of calibration standards) are detailed in Table B.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviation from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

11.4. Instrument start-up: The Ion Chromatography instrument and all of its components including the printer are routinely on and ready for operation. Though the system is on, the analyst has to initiate the following steps before the instrument is ready for analysis.

11.4.1. Ensure that the eluent is in sufficient supply for the entire run, and the waste carboy has plenty of capacity.

11.4.2. Through the Chromeleon software, access the control panel for the ICS-1000. Press the “start” button to initiate the instrument.

11.4.3. Make sure that the following instrument parameters are set:

- Pump Speed: 1.2 mL/min,
- Suppressor Current: 31 mA,
- Cell temperature: 30°C
- System Pressure: Pressure requirements vary for different columns. Follow the manufacturers recommended settings for each column.

11.4.4. Let the system equilibrate for 10 minutes. If the system pressure is fluctuating, follow the manufacture’s instructions for priming the pump. If this does not solve the problem, check the system for leaks. If no leaks are present, the pump head seals may need to be replaced.

11.4.5. The instrument is now ready for use.

11.5. Preliminary Set-up:

11.5.1. Establish the baseline by injecting at least one vial with reagent water through the system until a stable baseline is obtained.

11.5.2. Inject the calibration curve. Concentrations of the curve shall bracket the concentration range of the samples. Inject the lowest concentration first. Verify that the correlation coefficient is at least 0.995 for each anion of interest. Calibration is done monthly but is verified daily.

11.6. Sample Pre-treatment:

All samples and standards are filtered through a 0.2 micron filter that is part of the sample vial cap design prior to analysis. Samples that have particulates larger than 0.45 micron must be filtered before placing the aliquot in the sample vial. This will minimize pressure build up and dirt clogging the system.

Warning: Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disk to rupture during the process.

11.7. Sample screening: The conductivity of the samples is measured and used as a tool to determine approximate dilution needed to keep the sample reading within calibration range. Additionally, test strips for Chloride may be used to estimate the concentrations of this analyte. Follow manufacturer instructions for use, and prepare sample dilutions accordingly. Listed are the recommended dilutions to make based on the measured conductivity (EC):

Measured Conductivity (EC) [f] (umhos/cm)	Suggested Dilution [g] (XX)
1 to 1,000	1X
1,000 to 5,000	5X
5,000 to 10,000	10X
10,000 to 20,000	20X

[f] Approximate measurements only.
[g] Varying dilutions may be necessary toward the end of the run to ensure that the sample reading is at least 3-5 times higher than the lowest standard. Historical dilution data supersedes this table.

11.8. For holding time purposes and to minimize sample handling, sequential multiple dilutions per sample are highly recommended.

11.9. Check the pH of samples that have a very high EC reading to ensure that the samples are not preserved.

11.10. Samples that are viscous, have dark color, foul odor or known to have high salt content must be diluted immediately regardless of the EC reading to prevent instrument damage.

11.11. Soil samples are prepared by leaching one part sample with five parts of reagent water. Refer to SOP No. WS-WC-0049.

11.12. Sample analysis:

11.12.1. Verify system calibration daily with an ICV and, if required, re-calibrate.

11.12.2. For analytical sequence set-up/schedule, see Table B.

11.12.3. Samples must be homogenized prior to filtering and aliquoting.

11.12.4. Pour approximately 5 mL of standard or well mixed sample or dilution thereof into the sample vial, and cap.

11.12.5. Arrange sample vials into the cartridge to reflect the analytical order in the bench sheet following the order in Table B. Enter all the information in the Dionex Chromeleon software schedule.

11.12.6. Set-up the analytical schedule following the steps below:

11.12.6.1. From the Chromeleon browser screen, copy the last schedule and save it as a new file, using the normal date format. If an old curve is to be used, copy in the raw data files for the standards.

11.12.6.2. Enter the following information: Sample Name (Lot ID, Standard ID, etc), Dilution Factor, Program (i.e. AS22A Anions), Method (AS22A Anions), and Weight (only needed for DI leachates or soil samples).

11.12.6.3. After the last CCB, under SAMPLE NAME, enter “SHUTDOWN”; under PROGRAM, enter “ICS 1000 SHUTDOWN. This will enable the instrument to automatically load the shutdown method at the end of the run, and all components of ICS 1000 will be turned off.

11.12.7. When the schedule is built and samples are loaded into the autosampler, press the “run” on the autosampler , then ress the “play” button. This will initiate the run.

11.12.8. The instrument is now in analysis mode. Each injection will take approximately 10 minutes.

11.13. Confirmation by Coinjection:

In the event that a positive is not absolutely identifiable due to retention time shifts, a coinjection may be made, as follows:

11.13.1. Determine the optimum sample concentration for analysis (within 1/2 of the linear range).

11.13.2. Prepare a dilution if necessary. Add sample and spikes and bring to a final volume of 5mL. Gently mix, then analyze. The MS/MSD spike levels may be used. The neat sample final volume will be 5mL plus the spike volume.

11.13.3. Presence of merged/shouldering peaks is indicative of matrix effects rather than a positive for that anion.

11.14. Instrument Shut-down

11.14.1. Automatic shut-down - At the end of the schedule list, enter the shutdown method as part of the schedule or,

11.14.2. Manually shut the instrument down by pressing the "stop" button on the ICS1000 control panel.

11.14.3. Sample vials and acidic waste generated after the analysis must be disposed of according to the waste streams detailed in Section 15.

12. CALCULATIONS/DATA REDUCTION

12.1. Anion Identification

12.1.1. Identification occurs when a peak matching the retention time of the reference standard is found at a concentration above the reporting limit, or above the MDL if J flags are required.

12.1.2. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in a variation of retention times.

12.1.3. If manual integration is needed, refer to the Manual Integration Policy, QA-011-SAC. Save the file after manual integration.

12.1.4. The program Chromeleon marks a chromatogram that has been manually integrated by placing a [modified by *username*] above the chromatogram and an asterisk (*) next to the anion that was modified.

12.2. Confirmation

In some instances, a coinjection of standard solution and sample may be necessary to confirm the identity of a peak in the ion chromatogram. Details of preparing a coinjection analysis are discussed in Section 11.19

12.3. Calibration Range

12.3.1. If concentration of any analyte exceeds the working range as defined by the

calibration standards, then the sample must be diluted and reanalyzed for that analyte.

12.3.2. Responses for the diluted samples must be 3-5 times the level of the lowest standard.

12.3.3. It may be necessary to dilute samples due to matrix.

12.4. Calculations

All instrument concentrations are calculated using a linear regression calculation automatically performed by the instrument, according to the following equation:

Equation 1

Concentration (mg/L) $y=mx + b$

Where:

x and y are variables

b = intercept point of the regression line and the y axis

m = slope of the regression line

Equation 2

*Sample Concentration (mg/L) = Instrument Concentration (mg/L) * DF*

Where *DF* = Dilution Factor

Equation 3

*Sample Concentration (mg/kg) = Instrument Concentration (mg/L) * DF* * V_l / M_s

Where *DF* = Dilution Factor

V_l = Volume of Leachate (in L)

M_s = Mass of Soil (in kg)

NOTE: For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

12.5. Reporting Requirements

12.5.1. Reporting limits and units are described in Section 1.3.

12.5.2. Sample results are entered into a LIMS system in accordance with current QA policies. When reporting calculated percentages for the purposes of quality control, round to two significant digits unless the value is over 100%. If the value is over 100%, round to three significant digits.

12.5.3. Footnotes and anomalies when applicable must be included in data entry and data reduction process. Exceeded holding times must be immediately communicated to project managers and followed by electronically filed HTV.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit: The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be established annually and verified via an MDL check at 6 month intervals. See the MDL SOP WS-QA-0006 for details. The MDL must be below the reporting limit for each analyte.

13.3. Initial Demonstration: Each analyst must make a one time initial demonstration of capability for each individual method. This requires analysis of QC check samples containing all of the standard analytes for the method.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to one to three times the RL..

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4. Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.5. This method is recommended for use only by analysts experienced in the use of ion chromatography and in the interpretation of the ion chromatograms.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

15.1. Alkaline, neutral and/or acidic eluent waste generated by the analysis. The waste from the instrument is collected in 2.5 liter plastic coated jugs. When full, transfer the jug to the main waste area for shipment.

15.2. Contaminated solid waste such as IC syringes, filters, caps and empty vials utilized for sample preparation. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full or at the end of the day, whichever comes first, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Unused sample material. Pour any excess liquid into a 1-liter to 4-liter HPLC collection carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move the drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. EPA Test Method 300.0 entitled, “The Determination of Inorganic Anions by Ion Chromatography” published in EPA-600/R-93/100, Revision 2.1, August 1993.

16.2. SW-846 update III, Method 9056, Revision 0, September 1994

16.3. List of other SOPs cross-referenced in SOP.

16.3.1. QA Policy WS-PQA-003, Quality Control Program.

16.3.2. WS-QA-0006, Method Detection Limits and Instrument Detection Limits.

16.3.3. WS-WC-0049, Deionized Leaching Procedure For General Chemistry Analyses.

17. METHOD MODIFICATIONS

17.1. Deviations from reference method rationale:

17.1.1. This SOP covers anions listed on Part A of Method 300.0. This facility does not have the capability to perform analytes listed in Part B.

17.1.2. Eluent strength: The eluent strength used for this SOP has been changed from what is recommended in EPA Method 300.0 and SW-846 Method 9056,

respectively. The strength is changed to enhance peak area and height characteristic, baseline resolution and improve ion separation between analytes based on manufacturer's recommendation for the AS22A column.

17.1.3. Drying of Neat Materials: Section 7.5 of Method 300.0 is adopted with regards to drying of neat materials at 105 °C for at least 30 minutes instead of drying them at varying temperatures as Method 9056 recommends.

17.1.4. Neat Material Substitution: Section 7.5 of Method 300.0 and Section 5.5 of Method 9056 recommend using the following salts for stock standard solutions: NaBr, NaCl, and $NaNO_3$. This laboratory has substituted KBr, KCl, and KNO_3, respectively.

17.1.5. Stability of Standard Extended: Section 7.5 of Method 300.0 and Section 5.5 of Method 9056 both comment that stock standard solutions are valid for a period of one month if stored at 4°C, and that working solutions are valid for a period of one week (one day for nitrite and phosphate stocks). This laboratory maintains that stock standard solutions are valid for a period of one year when stored at 4°C, that Nitrite, Nitrate, and Ortho-phosphate solutions are stable for up to 6 months, and that all working standards are valid for a period of one month. The change of expiration dates are based on a stability study performed by the laboratory for these analyses.

17.1.6. Calibration utilizes linear regression with offset, set to include zero (0,0). This SOP calculates the concentration from the peak area using linear regression.

17.1.7. The columns specified in the reference method have been replaced with alternatives. This change was motivated by advances in technology which occurred after publication of the method. Likewise, the use of suppressor regenerant solution has been discontinued.

17.1.8. Nitrate + Nitrite determinations on preserved samples cannot be done by this method due to severe interferences introduced by the preservative. Samples for this determination are usually preserved with H_2SO_4 and should be analyzed by a different method such as the Cd-reduction/colorimetric method.

17.1.9. Preparation of diluted samples and standards using eluent as described in Section 7.2.1 of Method 9056 is not adopted here. For matrix matching purposes, all standard preparations and sample dilutions are made with reagent water so as not to bias the undiluted samples.

17.1.10. Section 10.4 of Method 300.0 recommends that the calibration be verified every 20 samples. This SOP requires that the calibration be verified every 10 injections.

17.1.11. Section 8.2 of Method 9056 specifies "After every 10 injections, analyze a mid-range calibration standard. If the instrument response has changed by more than 5%, recalibrate." This SOP incorporates this requirement in addition to the Method 300.0 ± 10% requirement when evaluating CCV standards.

17.1.12. Prep Factor for soil leaching procedure is changed from 10X (Section 11.7/Method 300.0) to 5X in order to meet lower reporting limits. The soil and water are mixed for 1 hour instead of 10 minutes, then centrifuged, decanted, and filtered.

17.1.13. LCS acceptance criteria for prep batch is 85-115 %, while the direct analysis batch is 90-110%. There is no guidance in either source method in regards to the LCS that is batched and prepared with soil/solid samples.

17.1.14. Section 9.3.1 of Method 300.0 criteria for Laboratory Reagent Blank (LRB) is below the MDL. Our criteria for Method Blank (MB) or LRB is below the reporting limit (RL), refer to QA PolicyWS-PQA-003.

17.1.15. Section 9.2.2 of Method 300.0 recommends that the Linear Calibration Range (LCR) must be determined initially and verified <u>every 6 months</u> or whenever a significant change in the instrument response (10% from Section 10.4) is observed or expected. This laboratory verifies the calibration with an ICV and reference standard prior to analysis, and mandates a new calibration <u>monthly</u>, or when eluent strength or eluent stock solutions are changed.

17.1.16. Section 7.3.4 of Method 300.0 - Calculation is done by linear regression with offset, set to include zero (0,0). The instrument automatically calculates concentration of the samples and is verified at the data reduction stage.

17.1.17. Section 8.3, 8.4/Method 9056 - Analyze one duplicate (DU) per 10 sample, one matrix spike (MS) every 20 samples. The laboratory adopted an MS/SD pair every 20 samples or less. Refer to WS-PQA-003, Quality Control Program.

17.1.18. Method 300.0 directs that MDLs should be performed semi-annually. TestAmericaSacramento's policy is to perform MDLs annually, with semi-annual verification.

18. ATTACHMENTS

18.1. Tables or figures referenced in body of SOP.

18.1.1. Table A - Quality Control and Calibration Criteria

18.1.2. Table B - Analytical Sequence (an example)

18.1.3. Table C - Instrument Operating Conditions

19. REVISION HISTORY

19.1. WS-WC-0009. Revision 3.3, Effective 07/18/2014

19.1.1. Inserted note following Section 10.3.4 - **NOTE:** Discrepancies may exist between the ICAL as evaluated using instrument software and the ICAL as evaluated using Chrom, resulting in the continued use of a failing ICAL.

The ICAL data in Chrom is to be evaluated by the end of the following business day and prior to starting a new analytical sequence to prevent the use of a failing ICAL.

19.1.2. Editorial changes.

19.2. WS-WC-0009, Revision 3.2, Defective 03/21/2014

19.2.1. Section 10.3.4 – Deleted ‘set to include zero (0,0).’

19.2.2. Section 12.4 – Deleted ‘set to include zero (0,0).’

19.2.3. Editorial changes.

19.3. WS-WC-0009, Revision 3.1, Effective 05/10/2013

19.3.1. Updated Table B to conform to TALS procedure.

19.3.2. Editorial changes.

19.4. WS-WC-0009, Revision 3, Effective 04/13/2012.

19.4.1. Clarification of ASTM Type II water.

19.4.2. Revision of calibration types.

19.4.3. Editorial revisions.

19.5. WS-WC-0009, Revision 2.6, Effective 02/17/2011.

19.5.1. Section 1.3: Updated RT’s and window sizes for anions.

19.5.2. Section 6: Updated P/N’s for guard columns and Anion Self Regenerating Suppressor.

19.5.3. Section 7.3: Updated eluent concentrations.

19.5.4. Updated Table C for new instrument parameters.

19.5.5. Section 10.3.3: Changed from weighted quadratic equation to weighted linear equation.

19.5.6. Section 12.4: Change from quadratic to linear equation.

19.5.7. Section 19.6.7: Changed from: “Linear ranges and detection limits are different from the method source and are based on instrument performance and historical limits.” to “Calculation is done by linear regression using peak area. This SOP calculates the concentration from the peak area using linear regression.”

Table A			
Acceptance Criteria and Corrective Actions-Anions			
QC Type	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Initial Calibration Curve	Calibrated initially, then monthly. Verified daily prior to analysis.	r > 0.995	Reanalyze once. If the problem persists, reprepare the standards, and recalibrate.
ICV/REF/LCS (Second Source Standard)	At start of every analytical sequence, following the initial calibration.	90%-110% Recovery	Reanalyze once. If the problem persists, reprepare the standards, reanalyze, and/or recalibrate.
ICB/CCB/MB	Directly following ICV/CCVs.	<Reporting Limit <½ RL for DoD QSM	Reanalyze once. If the problem persists, isolate the source of the problem and fix it. If the problem is isolated to the blank, reprepare, reanalyze and proceed. If the problem may have affected previous sample results (i.e. instrument failure, contaminated test tubes, etc.), reanalyze samples bracketed by the failed blank.
CCV	After every 10 samples and at the end of the analytical sequence.	90%-110% Recovery Method 9056: ± 5% RPD for adjacent CCV	Reanalyze once. If the problem persists, isolate the source of the problem and fix it. If the problem is isolated to the standard (i.e. misspike, scratched test tube, etc.), reprepare, reanalyze and proceed. If the problem may have affected previous sample results (i.e. instrument failure, contaminated test tubes, etc.), reanalyze samples bracketed by the failed standard.
MS/SD	1 MS/MSD pair per batch of 20 samples or fewer.	90%-110% Recovery, aqueous 80%-120% Recovery, Solids and wipes 20%RPD	Reanalyze once. If reanalysis recovery fails but % RPD passes, accept data. If reanalysis passes, report reanalysis.
MB-solid (ICB=MB for aqueous)	1 per batch of 20 samples or fewer	< Reporting limit <½ RL for DoD QSM	Reanalyze once. If problem persists, reprepare and reanalyze batch.
LCS - solid (ICV=LCS for aqueous)	1 per batch of 20 samples or fewer	85%-115%	Respike aliquot and reanalyze. If problem persists, reprepare and reanalyze batch.

Table B		
Analytical Sample Sequence (An example)		
Injection Number(s)	**Sample ID**	**Comments**
1	Reagent Blanks (Can be done manually and separately from the run.	Establish stable baseline.
2,3,4,5,6	Initial Calibration (Monthly, or when needed)	Least Concentrated to Most Concentrated Standard. (Not always needed, see text).
7	Initial Calibration Verification (ICV)	
8	Initial Calibration Blank (ICB)	Reagent water
9-18	Samples (including MS/SD if requested and prep MB/LCS and associated samples, MS/SD).	10 injections between ICV/ICB and CCV/CCB or CCV/CCB pairs..
19	Continuing Calibration Verification (CCV)	
20	Continuing Calibration Blank (CCB)	Use Level 0 Standard Solution. (Reagent water)
Repeat injections 9 through 20 until all samples are injected	10 injections followed by CCV/CCB.	Ending CCV/CCB closes the run
Last entry of the RUN	Enter as SHUTDOWN file in IC-1000 Program. SHUTDWN1 Program.pgm.	The instrument will automatically shut-off if the “shutdown method” is included in the scheduled run log.

Table C	
Instrument Operating Conditions, “IC6” Parameter under AS22 MethodHigh	
Parameter Name	**Parameter Value**
Minimum Area	**0.005**
Sensitivity	**0.0001**
Peak Slice	**10.00 s**
Fronting Sensitivity Factor	**2.5**
Tailing Sensitivity Factor	**5.5**
Minimum Width	**0.10 min**
Valley to Valley	**On**

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Title: Determination of Hexavalent Chromium By Manual Colorimetric Method [Method SW-846/7196A]

Approvals (Signature/Date):

Robert Hrabak 9/13/13 Date Technical Manager	Joe Schairer 10/7/13 Date Health & Safety Manager / Coordinator
Karla Buechler 9/13/13 Date Quality Assurance Manager	Zan Vicino for ZV 10/8/13 Date Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This procedure is used for the determination of dissolved hexavalent chromium by EPA method 7196A.

1.2. This method is applicable to waters, soils, sediments, solid waste samples, TCLP and STLC extracts.

1.3. The reporting limit is 0.01 mg/L for water samples and 0.05 mg/kg for soil and waste samples. The linear range of the calibration curve is 0.005 mg/L – 0.1 mg/L.

1.4. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically from the reaction with 1.5-diphenylcarbazide in acidic solution. The addition of an excess of diphenylcarbazide yields the red-violet product of unknown composition, and its absorbance is measured photometrically at 540 nm.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Hexavalent molybdenum and mercury salts react with diphenylcarbazide, but the red-violet intensity of the colors produced is much lower than that for chromium at the specified pH and concentrations up to 200 mg/L can be tolerated.

4.2. Vanadium interferes strongly at concentrations greater than 10 times the concentration of hexavalent chromium.

4.3. Iron at concentrations greater than 1 mg/L may produce a yellow color but should not interfere at the wavelength used in this analysis.

4.4. Turbidity and color in the samples may cause erroneously high absorbance readings. In such cases, filtration and or centrifugation may be required.

4.5. Reducing agents such as sulfite and organic compounds interfere by reducing hexavalent chromium to trivalent chromium.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide satisfactory protection.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in**

the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
1,5-Diphenylcarbazide	Irritant	None listed	Causes irritation to skin, eyes, mucous membranes, upper respiratory tract
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
ChromaVer 3 (Cr(VI) + Reagent)	Irritant	None established	Causes eye burns. May cause respiratory tract irritation.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 - TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. A ThermoSpectronic Genesys 20 spectrophotometer for use at 540 nm, providing a light path of 1 cm.

6.2. 1- cm disposable absorbance cell cuvettes.

6.3. 100 uL, 1 mL, 10 mL adjustable air displacement pipettes.

6.4. 13 mL capacity test tubes.

6.5. Narrow range pH test strips.

6.6. Miscellaneous laboratory glassware.

7. REAGENTS AND STANDARDS

7.1. Use reagent grade chemicals in all tests. "Certificates of Analysis" should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates when opened.

7.2. Reagent water is produced by a Millipore nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3. HACH ChromaVer 3 hexavalent chromium reagent.

7.4. Diphenylcarbazide (DPC) solution: Weigh and transfer 250 mg of 1,5-diphenylcarbazide into a 50 ml volumetric flack containing 25 ml of acetone. Dissolve and dilute to the mark with acetone. Store in a brown bottle. Do not use when solution becomes discolored.

7.5. Cr(VI) certified stock calibration standard, 1 mg/L: Obtain commercially. The expiration date for this standard is the earlier of the date printed on the container or one year after opening.

7.6. Cr(VI) certified stock second source standard, 10 mg/L: Obtain commercially and from a different manufacturer or with a different lot ID from the calibration stock solution (Section 7.5). The expiration date for this standard is the earlier of the date printed on the container or one year after opening.

7.7. 0.04 mg/L hexavalent chromium ICV/LCS standard: add 0.04 ml of 10 mg/L stock standard (Section 7.6) to a test tube and dilute to 10 mL with reagent water.

7.8. Reagent Grade 50% H_2SO_4.

7.9. Reagent Grade Acetone.

7.10. 1,5 Diphenylcarbazide powder.

7.11. Reagent grade potassium Permanganate ($KMnO_4$)

7.11.1. Potassium Permanganate Stock Solution: Dissolve 0.16 g of Potassium Permanganate into a 100 mL volumetric flask containing reagent water and fill to volume mark.

7.11.2. Potassium Permanganate, 0.10mM: Transfer 1.0 mL of Potassium

Permanganate Stock Solution into a 100 mL volumetric flask. Dilute to volume with reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The holding time for aqueous samples is 24 hours from collection.

8.2. For solids, the holding time to preparation (deionized water leaching in accordance with SOP WS-WC-0049) is 30 days, and the holding time to analysis is 24 hours and begins after extraction.

8.3. The holding time for leachates prepared in accordance with SOP WS-IP-0004 (e.g., TCLP, STLC, etc) is 24 hours from the end of the leaching procedure.

8.4. To retard the chemical activity of hexavalent chromium, aqueous and solid samples along with extracts must be stored at 4°C ± 2°C until analyzed.

9. QUALITY CONTROL

9.1. The wavelength accuracy of non-scanning (manually advanced) monochromators in VIS/UV-VIS spectrophotometers must be checked annually. The checks must be documented and available for inspection. Any repairs or problems are documented in the maintenance logbook for the spectrophotometer.

9.2. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be analyzed within the same time frame. A method blank (MB), laboratory control sample (LCS), or duplicate control sample (DCS) are analyzed as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD). An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples. Refer to the QC Program document (WS-PQA-0003) for more details.

9.3. One method blank (MB) must be reported for every 20 samples. If more than 20 samples are analyzed, an additional MB immediately preceding analysis of the second batch is reported as the MB for all samples associated with the second batch. For solid samples, a leachate blank is used as the MB. If a MB exceeds ± the reporting limit (RL) then all associated samples must be re-prepared and reanalyzed. For DOD projects, the MB must fall within ± ½ the RL. The exception is samples that are less than the RL and those that exceed 10X the concentration of the analyte in the MB. In such cases, the data can be reported and all corrective actions documented in a Nonconformance Memo. Refer to the QC Program document (WS-PQA-0003) for more details.

9.4. A laboratory control sample (LCS) must be reported for every 20 samples analyzed. If more than 20 samples are analyzed, an additional LCS immediately preceding analysis of the second batch of samples is reported as the LCS for all samples associated with the second batch. If a LCSD is required, a second LCS is analyzed and reported. The leachate MB is spiked and reported as the LCS for solid samples. The LCS acceptance criterion is 90-110% recovery. If the LCS fails high and the samples are below the RL, the data can be reported and all corrective actions documented in a Nonconformance Memo. If the LCS fails low then all associated samples must be re-prepared and reanalyzed. Certain programs, such as DOD QSM, will not accept any failures (including high with non-detect samples) and require repreparation/reanalysis. Refer to the QC Program document (WS-PQA-0003) for more details.

9.5. A matrix spike/matrix spike duplicate (MS/MSD) pair must be analyzed with every batch. If the batch is greater than 10 samples, a second MS/MSD pair is required. MS/MSD pairs are aliquots of a selected field sample spiked with analyte of known identity and concentration. Spiked samples with percent recoveries or precision (RPD) outside of designated control limits for an analyte must be associated with a LCS that demonstrates that the system is in control. The MS/MSD acceptance criterion is 85-115% recovery. Carefully evaluate and review any out-of-control event. Re-analysis of an MS/MSD pair may be required if there is any question that anomalous results may be due to instrument problems and not the matrix of the sample.

Note: Samples identified as field blanks, equipment blanks, or trip blanks should not be used for MS/SD analysis.

9.5.1. Recovery trends of less than 85% should be assessed to determine if the low spike recovery is due to the presence of residual reducing agent or suppressive interference. Contact the Project Manager to determine how to proceed and document all corrective actions as appropriate in an Nonconformance Memo if low recovery persists after the following assessment. Refer to the QC Program document (WS-PQA-0003) for more details.

9.5.1.1. If the sample has produced an acidic extract, the presence of residual reducing agent is determined by taking a fresh aliquot of the sample or extract and adjusting the pH to 8.0-8.5 using 1 N sodium hydroxide, then spiking and analyzing. The data can be reported if the recovery is acceptable. Document all corrective actions in an Nonconformance Memo.

9.5.1.2. If the sample has not produced an acidic extract and suppressive interference is indicated, a fresh aliquot of the sample or extract is diluted, spiked and analyzed. The data can be reported if the recovery is acceptable. Document all corrective actions in an Nonconformance Memo.

9.6. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a sample/sample duplicate or MS/SD pair or as required by client or regulatory requirements. The LCS and LCSD are evaluated independently for acceptance. Refer to the QC Program document (WS-PQA-0003) for more details.

9.7. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard immediately following instrument calibration. This standard must be at a concentration different from the CCV standard. The ICV must fall within ± 10% of the true value of the standard solution. An ICB prepared the same as the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within ± the RL from zero. For DOD projects, the ICB must fall within ± ½ the RL. If either the ICV or ICB fail to meet acceptance criteria, the analysis must be terminated, the problem corrected, and the instrument re-calibrated.

9.8. Continuing Calibration Verification (CCV/CCB) – Calibration accuracy is evaluated throughout the analytical run by analyzing a continuing calibration verification standard (CCV) of a known concentration every 10 samples. In this case, a sample is any analysis that registers a result, whether the result is reported or not. The CCV must be a mid-range standard at a concentration other than that of the ICV. In most cases, the CCV is one of the middle standards used during calibration. The CCV must fall within ± 10% of the true value. A continuing calibration blank (CCB) prepared the same as the calibration blank must be analyzed following every CCV. The CCB must fall within ± RL from zero. For DOD projects, the CCB must fall within ± ½ the RL. Each CCV and CCB analyzed must reflect the conditions of the analysis of the associated samples. If a CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument re-calibrated, the calibration verified and the affected samples reanalyzed.

10. CALIBRATION

10.1. Annual Spectrophotometer Wavelength Check – The wavelength accuracy of non-scanning (manually advanced) monochromators in VIS/UV-VIS spectrophotometers is checked annually. The wavelengths of two peak maxima of a dilute solution of Potassium Permanganate (0.1mM $KMnO_4$) are determined on the VIS/UV-VVIS spectrophotometer and these determined wavelengths are compared to the true values.

10.1.1. Allow the spectrophotometer to warm up for at least ½ hour before using.

10.1.2. Set the monochromator to wavelength of 516 nm and zero the instrument with reagent water. Fill a test tube with 0.10mM $KMnO_4$ solution, insert into the instrument and record the absorbance.

10.1.3. Advance the monochromator by a wavelength increment of 5 nm, zero the instrument with reagent water and record the absorbance of the 0.10mM

$KMnO_4$ solution. Continue advancing the monochromator in 5 nm increments to 556 nm, zeroing, and recording the absorbance of the 0.10mM $KMnO_4$ solution at each wavelength.

10.1.4. Find and record the instrument's wavelengths of maximum absorbance from the tabulation of absorbance values. One maximum absorbance should occur at 526 nm and a second at either 541 nm or 546 nm. If these criteria are not met, the instrument should be serviced by qualified personnel.

10.2. Linear Dynamic Range Verification (LDR) – The linear dynamic range must be verified every six months for each analyte wavelength used on each instrument. The linear dynamic range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear dynamic range limit must be analyzed during a routine analytical run. For the initial determination and semi-annual verification of the upper limit of the LDR for each wavelength, determine the signal responses from a minimum of three different concentration standards across the estimated range. One standard should be at or near the upper limit of the estimated range. The concentration measured at the upper limit of the LDR should be no more than 10% less than the expected level extrapolated from lower standards New dynamic ranges must be determined if the instrument is adjusted in any way that may affect the LDRs.. The LDR data must be documented and kept on file..

10.3. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.4. Prepare the calibration curve every 24 hours of operation.

10.5. One standard must be analyzed at the reporting limit. Analyze standards in ascending order beginning with the calibration blank.

10.5.1. Prepare the working hexavalent chromium calibration standards by diluting the 1.0 mg/L Cr(VI) stock calibration standard with reagent water as follows:

Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	0.05	10	0.005
2	0.1	10	0.01
3	0.25	10	0.025
4	0.5	10	0.05
5	1.0	10	0.1

10.5.2. The 0.005 mg/L Cr(VI) is analyzed as the first standard to meet client-specific requirements.

10.5.3. An ICV/LCS of 0.04 mg/L Cr(VI) is made by diluting 0.04 mL of the 10.0 mg/L Cr(VI) second source standard to 10 mL using reagent water in a 13 mL

test tube.

10.5.4. The 0.05 mg/L Cr(VI) standard is also used as the CCV.

10.6. The calibration curve must have a correlation coefficient (r) ≥ 0.995.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance Memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance Memo shall be used for this documentation.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described (see WS-QA-0023, Nonconformance and Corrective Action System).

11.3. This analytical method is restricted to use by, or under the supervision of, an analyst experienced in the operation of a manual spectrophotometer and the interpretation of its results.

11.4. Allow the spectrophotometer to warm up for at least 1/2 hour before using.

11.5. Sample and Standard Analysis

11.5.1. Prepare MS/MSDs for aqueous samples by spiking 10 mL of sample with 0.05 mL of the 10.0 mg/L Cr(VI) stock second source calibration standard. The resulting spiking level is 0.05 mg/L Cr(VI).

11.5.2. Extract solid samples at 10.0 g solids to 50 g reagent water as directed in SOP WS-WC-0049.

11.5.2.1. Prepare LCSs for leachates by spiking the leached method blank with 0.04 mL of the 10 mg/L Cr(VI) stock second source calibration standard and diluting to a final volume of 10 mL with method blank solution. The final concentration of the LCS is 0.2 mg/kg.

11.5.2.2. Prepare MS/MSDs for leachates by spiking 10 mL of a leached sample with 0.05 mL of the 10.0 mg/L Cr(VI) stock second source

calibration standard. If a sample requires dilution due to matrix or high analyte levels, the appropriate volume of sample is spiked with 0.05 mL of the 10.0 mg/L Cr(VI) stock second source calibration standard and diluted to a final volume of 10 mL with reagent water. The final concentration of the leachate is 0.25 mg/kg.

11.5.3. Make the calibration curve, ICV, and CCVs according to Section 10.

11.5.4. Transfer 10 mL of each sample into two test tubes, one to be measured for any background absorbance (color blank), and the other to be measured for color development (sample Cr(VI) absorbance).

11.5.5. If the samples are turbid, filter or centrifuge prior to the addition of reagents.

11.5.6. For calibration standards, ICVs, CCVs, and all blanks, making a color blank for background absorbance is not necessary.

11.5.7. Add the contents of one ChromaVer 3 hexavalent chromium reagent pillow or 0.2 ml of DPC solution to each of the sample and standard tubes. If using DPC solution, also add one drop of 50% H_2SO_4. Do not add the DPC reagent to the background absorbance tubes.

11.5.8. Add one drop of 50% H_2SO_4 to each color blank.

11.5.9. Cover and mix well. Allow 5-10 minutes for complete color development.

11.5.10. Check the pH of each color sample to assure the pH is 2 ± 0.5. Samples with high buffering capacity may interfere with obtaining the desired pH using the ChromaVer 3 reagent alone. If this occurs, add 50% H_2SO_4 to reach the desired pH.

11.5.11. Use reagent water to zero the instrument absorbance reading at 540 nm.

11.5.12. Pour an aliquot into a disposable absorbance cuvette and insert into the spectrophotometer, assuring the clear windows are directed towards the pathlength.

11.5.13. Read the absorbance of the calibration curve, including a calibration blank treated the same as all of the treated samples and standards.

11.5.14. Read the background absorbance of the color blank sample followed by the reading of the treated sample.

11.5.15. Enter the results in “real time” into the hexavalent chromium linear regression computer program.

12. CALCULATIONS/DATA REDUCTION

12.1. Data is calculated using linear regression.

12.2. The absorbance reading for all samples is corrected using the following calculation:

12.2.1. (Color Sample Absorbance – Background Absorbance) = True Absorbance

12.2.2. The sample concentration is calculated by plotting the true absorbance reading against the standard curve.

12.3. Samples falling outside the linear range must be diluted and reanalyzed. The Dilution Factor (DF) is used to calculate the final concentration of the diluted sample.

12.4. For soil leachates, calculate the final concentration in by multiplying the prep factor (PF) by the final concentration found in the leachate in mg/L. Final units are mg/kg.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be 1-4 times the RL.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated

failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acidic liquid waste generated by the analysis is collected in a 1L polymer jar. When the polymer jar is full, or after no more than one year, move the carboy to the waste collection area where it will be consolidated into a plastic LLE drum for shipment.

15.2. Contaminated disposable glass or plastic test tubes and cuvettes. Collect the liquid waste as described above, then drop the glass into a contaminated glass box. When the box is full or after no more than 75 days, move it to the waste collection area for shipment. Dispose of plastic test tubes and cuvettes in the appropriate 5-gallon trash bucket with lid for lab trash to be incinerated. When the bucket if full or after no more than 1 year, move it to a 55-gallon drum for lab trash to be incinerated. When the 55-gallon drum is full or after no more than 90 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Method 7196A, "Chromium, Hexavalent (Colorimetric)", Test Methods For Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, Final Update II; EPA July 1992.

16.2. HACH, Method 8023.

16.3. Handbook for Analytical Quality Control in Water and Wastewater laboratories, EPA-600/4, 79-019, U.S. EPA Office of Research and Development, Cincinnati March 1979.

16.4. Environmental Laboratory Approval Program Certification Manual; New York DOH, Albany, NY.

17. METHOD MODIFICATIONS

17.1. Reduced volume versions of the method source applied to this SOP are acceptable as long as the same volume-ratio of samples and reagents are used and they meet the quality control performance requirements stated in the method.

17.2. Section 7.3.4 of the reference Method is not included in this SOP. This facility does not perform methods 7195 or 7197.

17.3. HACH *ChromaVer 3* hexavalent chromium reagent can be used as a replacement for diphenylcarbazide and H_2SO_4.

17.4. pH strips are used instead of a pH meter to measure and adjust sample pH.

17.5. Sections 7.5 and 8.6 of the reference method (method of standard addition for delisting petitions) are not included in this SOP. This procedure is not appropriate for delisting petition.

18. ATTACHMENTS

18.1. None.

19. REVISION HISTORY

19.1. WS-WC-0020, Revision 7.5, Effective 10/21/2013

19.1.1. Section 4.1, clarified that the pH aids in tolerance of the interference

19.1.2. Section 5.2, added Chromver reagent to the table.

19.1.3. .Sections 7.5 and 7.6, clarified expiration.

19.1.4. Section 9, removed the use of the ICB and ICV as the MB and LCS, respectively. Similarly, the CCB and CCV are no longer used as MB or LCS.

19.1.5. Section 9.5, added acceptance criteria statement.

19.1.6. Section 9.5.1, added corrective actions for MS/MSD failure.

19.1.7. Section 10.1, corrected units from mm to nm.

19.1.8. Editorial changes.

19.2. WS-WC-0020, Revision 7.4, Effective 6/08/2012

19.2.1. Updated annual spectrophotometer wavelength check procedures in

Sections7 and 10.

19.2.2. Added Potassium Permanganate to Section 7.

19.2.3. Editorial revision.

19.3. WS-WC-0020, Revision 7.3, Effective 2/13/2011

19.3.1. Modified Section 6.1 to read: “A ThermoSpectronic Genesys 20 spectrophotometer for use at 540 nm, providing a light path of 1 cm.”

19.3.2. Editorial Revisions

19.3.3. Switched order of Sections 9.4.1 and 9.4.2.

19.4. WS-WC-0020, Revision 7.2, Effective 9/4/2009

19.4.1. Changed SOP header from [Method SW-946/7196A] to [Method SW-846/7196A].

19.5. WS-WC-0020, Revision 7.1, Effective 9/4/2009

19.5.1. Added Section 1.4, “When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.”

19.5.2. Inserted Section 10.1, “For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.”

19.6. WS-WC-0020, Revision 7, Effective 8/29/2008

19.6.1. Updated to TestAmerica format.

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Title: Determination of Alkalinity, Conductivity, and pH [Methods: SM 4500 H^+ B, SM 2320B, EPA 9040B, EPA 9040C w/ Update IV, EPA 120.1, SM2510B]

Approvals (Signature/Date):

3/25/14 3/25/14 TP 3/25/14 Thep Phomsopha — Date Inorganic Supervisor	3/18/14 Joe Schairer — Date Health & Safety Manager / Coordinator
3/26/2014 Lisa Stafford — Date Interim Quality Assurance Manager	3/28/14 Robert Hrabak — Date Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This procedure defines the automated analysis of alkalinity (SM 2320B), pH (SM 4500 H^+ B, 9040B, and 9040C w/ Update IV), and conductivity (EPA Method 120.1, SM2510B). The instrument used for analysis is a Man-Tech PC-Titration Plus system equipped with PC-Titrate software and a standard PC-Titrate XYZ autosampler. If the autotitrator is malfunctioning or in the case of short hold aqueous samples, manual pH measurement can be performed following procedure in SOP WS-WC-0044. The different forms of alkalinity (total, bicarbonate, carbonate, and hydroxide) can be calculated.

1.2. The detection limits are:

pH --Detection limit not applicable, range 2-12 pH units.
Specific conductance – 2 µmhos/cm
Alkalinity – 5 mg/L as calcium carbonate (25 mg/kg for solids)

1.3. This method is applicable to drinking, surface and saline waters, domestic and industrial wastes. Solid samples require a leaching process prior to analysis (see SOP WS-WC-0049). Soil samples for pH by method 9045D w/ Update IV are analyzed according to SOP WS-WC-0044.

1.4. This method is applicable to all ranges likely to be encountered. As a practical matter, samples with alkalinities greater than 1500 mg/L as calcium carbonate should be analyzed using a stronger titrant. For lower detection limits, analyze the sample using 0.02N H_2SO_4 acid as the titrant.

2. SUMMARY OF METHOD

2.1. Samples are analyzed on an automatic titrator. The instrument is capable of analyzing any or all of the components defined in Section 1.1 during a single analytical run.

2.2. Specific conductance is measured by application of a voltage across the conductivity cell in series with a known reference resistance. The ratio of voltage across the cell and the known resistance is measured which allows the resistance of the cell to be calculated. This is then converted to the conductance of the cell. By calibrating against a solution of known conductance, the cell constant is determined and also the specific conductance of the sample in the cell. A thermocouple measures the temperature of the sample and electronically corrects the measurements to 25°C.

2.3. The pH is determined electrometrically with a glass electrode in combination with a reference electrode. The special glass used in the electrode develops a voltage across it that depends on the pH of the solution being analyzed. The voltage is measured and converted to pH by calibration against buffers of known pH.

2.4. Alkalinity is determined by titrating the sample with a standardized acid to specified endpoints (pH 8.3 and 4.5). Alkalinity is calculated from the volume of acid required to reach the endpoints and is traditionally reported as mg/L calcium carbonate.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. pH Interferences

4.1.1. The pH response of most glass electrodes is imperfect at both ends of the scale. The indicated pH value of highly alkaline solutions, as measured with the glass electrode, will be too low. The indicated pH value of salts and strong acids having a pH less than 1 will often be higher than the true pH value. Interferences can be minimized by the selection of the proper electrodes for these conditions. For example, sodium may interfere at pH > 10, and is controlled by using a “low sodium error” electrode.

4.1.2. The glass electrode, in general is not subject to solution interference from color, turbidity, colloidal material, oxidants, reductants, or high salinity.

4.1.3. Coatings of oily material or particulate matter can impair electrode response. These coatings can generally be removed with gentle wiping or washing with a mild detergent.

4.1.4. Temperature variations will change the pH of the samples and also affect electrode response. Electronic temperature correction must be used to correct for electrode response.

4.2. Alkalinity Interference

4.2.1. Salts of weak organic and inorganic acids will contribute to alkalinity. If the alkalinity is intended to be a measure of carbonate and bicarbonate only, the presence of these substances will cause high results.

4.2.2. See Section 4.1.3 for the effect of oily material on the pH electrode.

4.2.3. Particulates in the sample may affect the alkalinity results.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, nitrile, and vinyl gloves provide adequate protection against chemicals and reagents typically used with this procedure

5.1.2. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.3. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Materials Section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid (1)	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Sodium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydradator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Total Ionic Strength Buffer (Acetic acid content)	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. The Man-Tech PC Titration Plus system consists of six major components. An autosampler, a Burivar I/2 burette for accurate delivery of titrant, a TitraSip module for titration, PC-Tis interface to the PC, a conductivity meter, and the titration/reaction vessel with stirring mechanism.

6.1.1. There are two electrodes used by the instrument. A conductivity electrode is mounted on the robotic arm of the autosampler to measure the conductivity in the autosampler tube. A pH electrode is installed into a titration vessel where the sample to be analyzed is transferred via the TitraSip module. The addition of reagents, and analyses of pH, and alkalinity take place within the titration vessel.

6.1.2. Recommended pH probe is ManTech PCD-80-PH1200.

6.2. Conductivity electrode.

6.3. Model 4310 conductivity meter with auto range from 0.01 umhos/cm to 199.9 mmhos/cm.

6.4. 50 mL polypropylene tubes.

6.5. Miscellaneous laboratory apparatus and glassware.

7. REAGENTS AND STANDARDS

7.1. Reagent water is produced by a Millipore Nanopure system. Water shall not contain particles larger than 0.20 micron and have a resistance of at least 1 Mohm/cm, or equivalently, a conductivity no greater than 1 µmhos/cm. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks, in accordance with SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water.

7.2. pH Reagents and chemicals

7.2.1. Certified buffer solutions: 2.0, 4.0, 7.0, 8.0, 10.0 and 12.0.

7.2.2. Alternate source as an ICV or reference check. This must be obtained from a separate vendor.

7.2.3. 3N KCl pH probe reference solution.

7.3. Alkalinity Reagents and chemicals

7.3.1. Certified Stock Standards: ICV and CCV solutions are procured as QC solutions from separate vendors. The true values change as different mixed certified stocks are received.

7.3.2. 0.02N Certified sulfuric acid solution: obtained with certificate of analysis from a defined vendor.

Note: Alkalinity measurements that exceed 1500 mg$CaCO_3$/L require analysis using a 0.1N certified stock sulfuric acid solution.

7.3.3. pH buffer solutions: 2.0, 4.0, 7.0, 8.0, 10.0 and 12.0.

7.4. Conductivity Reagents and Chemicals

7.4.1. Certified conductivity standards are used to calibrate the meter. In most cases, the standards are 1413 umhos/cm at 25ºC. Fresh aliquots of the standard must be used for calibration.

7.4.2. Certified conductivity standards of varying concentration are obtained from separate vendors for use in ICV/CCV analysis. Fresh aliquots of the standard must be used for analysis.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are to be collected in plastic bottles and refrigerated to 4°C.

8.2. Samples for alkalinity must not be filtered, diluted, concentrated, or altered in any way.

8.3. Upon arrival to the laboratory, pH samples must be directly delivered to an analyst in the general chemistry area for immediate analysis. The official holding times for these parameters are: pH – 15 minutes; Conductance – 28 days; Alkalinity – 14 days.

9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents, and standards. A batch must be analyzed within the same time frame. A Method Blank (MB) and Laboratory Control Sample (LCS) are analyzed as a part of every batch. Each batch must also be processed with a Sample Duplicate (DU). An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples (see policy WS-PQA-003 (Quality Control Program) for more details).

9.2. One Method Blank (MB) must be reported for every 20 samples, with the exception of pH, which has no MB analysis. If a MB exceeds ± the Reporting Limit (RL) than the samples must be re-extracted. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the MB. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance Memo. See policy WS-PQA-003 (Quality Control Program) for more details.

9.3. If more than 20 samples are analyzed, an additional MB immediately preceding analysis of the second batch is reported as the MB for all samples associated with the batch. If a MB exceeds +/- the RL, the analysis must be stopped, the problem resolved, and the samples associated with the out of control MB must be re-analyzed. The 20 sample batch criteria excludes Sample Duplicate analyses (see policy WS-PQA-003 (Quality Control Program) for further details).

9.4. A Laboratory Control Sample (LCS) must be reported for every 20 samples analyzed. If more than 20 samples are analyzed, an additional LCS immediately preceding analysis of the second batch of samples is reported as the LCS for the associated samples. If a Laboratory Control Sample Duplicate (LCSD or DCS) is required, an

additional aliquot of the LCS solution is analyzed as the LCSD. As with ICV/CCVs, the LCS acceptance criterion is 90-110% recovery with the exception of pH. The pH reading must be within 0.1 pH units of the true value.

9.5. For alkalinity, pH, and conductivity, a Sample and Sample Duplicate (DU) are analyzed in place of an MS/MSD pair in association with every batch. A Sample/ Sample Duplicate are replicate samples set up in separate autosampler tubes and analyzed within a single analytical run. The relative percent difference (RPD) between the Sample and the Sample Duplicate must be within 20%. For pH, the measurement between the Sample and Sample Duplicate cannot exceed 0.1 pH units. For the tests defined in this SOP, careful evaluation of Sample/Sample Duplicate results must be considered when differences are significant since these tests exhibit good accuracy.

9.6. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for Sample/Sample Duplicate analysis.

9.7. A Laboratory Control Sample Duplicate (LCS/LCSD) may be substituted when insufficient volume is provided to process a Sample/Sample Duplicate if required by client or program. The LCS and LCSD are evaluated independently for acceptance (see policy WS-PQA-003 (Quality Control Program) for further details).

9.8. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard, the ICV. This standard must be at a concentration different from those used to calibrate the instrument and different from the CCV standard. The ICV must fall within +/- 10% of the true value of the standard solution. For pH the value must be +/- 0.1 pH units. An ICB prepared the same as the calibration blank, or from nanopure water, must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet acceptance criteria, the analysis must be terminated, the problem corrected, and the instrument re-calibrated.

Note: pH has no ICB or CCB analysis.

9.9. Continuing Calibration Verification (CCV/CCB) – Calibration accuracy is analyzed throughout the analytical run by analyzing a CCV of a known concentration every 10 samples. In this case a sample is any analysis that registers a result, whether the result is reported or not. The CCV must be a mid-range standard at a concentration other than that of the ICV. In most cases the CCV is one of the middle standards used during calibration. The CCV must fall within +/- 10% of the true value. For pH the value must be +/- 0.1 pH units. A CCB prepared the same as the calibration standard must be analyzed following every CCV. The CCB must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of the analysis of the associated samples. Do not run rinses or increase rinse times prior to CCVs unless you do so between every sample. Sample results may only be reported when bracketed by valid

ICV/ICB and or CCV/CCB pairs as applicable. If a CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument re-calibrated, the calibration verified, and the affected samples reanalyzed.

10. CALIBRATION

10.1. Calibration of the instrument must be performed every 24 hours of operation.

10.2. Calibrate according to the instrument manufacturer's instructions. Analyze standards in ascending order.

NOTE: Refer to the following sections in the instrument manufacturers handbook for additional information. Section 13.10.5 (pH and alkalinity), and Section 13.12.5 (conductivity).

10.3. The calibration curve must have a correlation coefficient of > 0.995 for pH, or the instrument must be stopped and re-calibrated prior to running samples.

10.4. The accuracy of the calibration is verified using a secondary source (ICV). See Section 9.8 for more details.

10.5. The instrument must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart.

10.6. Burette accuracy is verified annually. Acceptance criteria is %CV is less than or equal to 0.2%. (CV = standard deviation of 10 measurements/average of 10 measurements.) See Table 1 for an example. File is also stored on the public drive under Genchem/AT2 Burette Syringe Calibration.

10.7. The sample dosing pump accuracy is verified annually. However, based on analysts discretion, this accuracy may be verified more frequently if the analyst suspects that the dosing pump is in need of adjustment based on data evaluation. To determine the pump flow rate, select the PUMP CAL icon in the PC-Titrate software. It is located in the lower left corner of the display screen. The software automatically prompts the analyst once the icon is selected. Follow the instructions. In order to enter the flow rate into the software program, follow this path: Interface → Hardware Set-Up → Digital/Amplifiers. Enter the flow rate that was determined into the dosing meter pump cell. This flow rate is used by the software to calculate the amount of time required to deliver 25ml of sample to the mixing chamber.

10.8. All pH calculations are temperature compensated. In order to view this in the software, follow this path: Setup → Analysis Schedule → Load → Conductivity/pH → Click on the second "Assign UDV" in the schedule table → Click on EDIT icon. This will show that the Thermistor is checked for temperature compensation. The Thermistor is verified annually.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo (NCM)and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The NCM will be filed in the project file. Any deviations from this procedure identified after the work has been completed must be documented as a Non-Conformance, with a cause and corrective action described. A NCM shall be used for this documentation.

11.2. Obtain samples from sample receiving. Check each sample for special instructions. Thoroughly homogenize samples before taking an aliquot for analysis. Allow aliquots to equilibrate to room temperature before analyzing.

11.3. Pre-run Checklist

11.3.1. Assure the nanopure water reservoir is full.

11.3.2. For alkalinity, assure the storage bottle contains enough 0.02 N H_2SO_4 to complete the analysis.

11.3.3. For alkalinity assure that no gas bubbles are present in the I/2 Burette module. If gas has accumulated in the burette, go to the Maintenance Section of the operations manual for the degassing procedure.

11.3.4. Assure that the pH electrode is filled with the appropriate electrolyte solution.

11.3.5. Assure that the waste carboy has adequate space for the run to complete without overfilling.

11.4. Starting the analysis

11.4.1. Templates are pre-programmed into the instrument for various combinations of analyses. Each template contains all of the parameters required to operate the system for analysis of the chosen analytes.

11.4.2. The template contains the run sequences with pre-programmed calibration commands and instrument QC locations.

11.4.3. Program sample IDs into autosampler positions in the template and save the

file.

11.4.4. Place calibration solutions, instrument QC and samples into the appropriate positions and start the analysis.

11.4.5. For conductivity, the meter has to be calibrated prior to initiation of the analysis.

11.4.6. At the end of any run sequence, a storage solution has to be poured into the position following the last analysis. The instrument automatically goes to this position to fill the reservoir for periods of non-operation.

11.5. pH analysis

11.5.1. Quantitative results can only be reported between 2 and 12 pH units per the calibration range.

11.5.2. Make sure the pH probe is filled with 3N KCl solution. The refill procedure is described in the PC-Titrate Hardware Guide.

11.5.3. For pH, the sample is transferred from a sipper, through the TitraSip module to the titration vessel.

11.5.4. Set up the complete run sequence for the buffer solutions and samples to be analyzed.

11.5.5. Pour approximately 40-45 mL of calibration buffer and samples into sample tubes and calibrate the instrument using pH buffers 2, 4, 7, 10, and 12.

11.5.6. Begin the analysis with an ICV at pH 8.0. There is no ICB analysis for pH.

11.5.7. The vessel is automatically rinsed with DI water between each analysis.

11.6. Alkalinity analysis

11.6.1. An unaltered sample is titrated to an end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.

11.6.2. The total alkalinity measurement can be broken down to the measurement of carbonate, bicarbonate, and hydroxide alkalinity.

11.6.3. Set up and calibrate the instrument for pH according to steps in Section 11.6.5.

11.6.4. For alkalinity, the sample is transferred from a sipper, through the TitraSip module to the titration vessel.

11.6.5. 0.02N sulfuric acid is added via the Burivar I/2 burette until the solution reaches a pH of 4.5. The precision of the burette is +/-0.2%.

11.6.6. Begin the analysis with an ICV/ICB.

11.6.7. For solid samples, a 10 mL aliquot of the DI leach is used for analysis.

Note: Alkalinity measurements that exceed 1500 mg $CaCO_3$/L require analysis using 0.1N certified stock sulfuric acid solution.

11.7. Conductivity Analysis

11.7.1. Samples are measured by a conductivity probe attached directly to the robotic arm of the autosampler and the conductivity is measured in the 50 mL sample tubes.

11.7.2. Results are automatically corrected to 25ºC with a predefined temperature coefficient of 1.91%.

11.7.3. The electrode is fragile and must be handled carefully. The electrode should be stored submersed in water.

11.7.4. The reporting limit for conductivity is 2.0 umhos/cm (2.0 uS/cm).

11.7.5. Calibrate the conductivity meter using a single certified standard programmed into the conductivity meter. The autosampler is not used to calibrate the conductivity meter.

11.7.6. Pouring 40-45 mL of sample into each tube, load the autosampler and begin analysis, starting with an ICV/ICB pair.

11.8. No specific conversion is needed for this instrument. The conductivity meter automatically carries out temperature compensation. The reference temperature is preset at 25ºC and the temperature coefficient is set at 1.91%.

12. CALCULATIONS/DATA REDUCTION

12.1. Alkalinity Calculation (performed by software)

12.1.1. Alkalinity of samples:

(Normality of acid) x (mL of acid) x 50,000 / Sample volume (mL)

12.1.2. Alkalinity of low samples (<20 ppm):

[(2A – B) x Normality of acid x 50,000 / Sample volume (mL)

A = ml of acid to pH 4.5 B = mL of acid to pH 4.2

12.1.3. Alkalinity in Solids

$$Alkalinity\ in\ solids(mg/g) = Alkalinity(mg/L)x\frac{FV(mL)}{IW(g)}$$

FV = Final Volume
IW= Initial Weight

12.1.4. Alkalinity equilibrium between the components of the alkalinity buffer system are calculated by the instrument software according to the following:

Result of Titration	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
P = ND	ND	ND	T
P < T/2	ND	2P	T - 2P
P = T/2	ND	2P	ND
P > T/2	2P - T	2(T - P)	ND
P = T	T	ND	ND

Where:

T = Total Alkalinity = Alkalinity at pH 4.5

P = Phenolphthalein Alkalinity = Alkalinity at pH 8.3

12.1.5. Default sample volume for aqueous analysis is 20 mL. Default sample volume for solid analysis is 10 mL, taken from DI leachate.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that an associate who has been properly trained in its use and has the required expertise performs this procedure.

13.2. Both prep and analytical chemists must pass the initial demonstration of capability (IDOC), as outlined by this facility.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out:

15.1. Aqueous alkaline or acidic waste generated by the analysis. This is collected in a 5-gallon plastic carboy. When the carboy is full, or after no more than one year, the pH should be checked. If not neutral or slightly alkaline, add a small amount of sodium bicarbonate to the waste to neutralize it at the fume hood. Then move the carboy to the waste collection area where it will be consolidated into a plastic LLE drum for shipment.

15.2. Contaminated disposable glass and/or plastic materials used in the analysis. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, or after no more than one year, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Methods for Chemical Analysis of Water and Wastes, Method 150.1, pH Method 150.1 (Electrometric), (Revised 1982).

16.2. Methods for Chemical Analysis of Water and Wastes, Method 120.1, Conductance, Method 120.1 (Specific Conductance, µmhos at 25˚C), (Revised 1982).

16.3. Methods for Chemical Analysis of Water and Wastes", Method 310.1, Alkalinity Method 310.1 (Titrimetric, pH 4.5), (Revised 1978).

16.4. Standard Methods for the Examination of Water and Wastewater, Method 2320B (Alkalinity, Titration Method), 18th Edition, 1992.

16.5. Standard Methods for the Examination of Water and Wastewater, Method 2320B (Alkalinity, Titration Method), 19th Edition, 1995.

16.6. Method 2320B, Alkalinity – Titration Method, "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.

16.7. Standard Methods for the Examination of Water and Wastewater, Method 2510B (Conductivity, Laboratory Method), 18th Edition, 1992.

16.8. Standard Methods for the Examination of Water and Wastewater, Method 2510B (Conductivity, Laboratory Method), 19th Edition, 1995.

16.9. Method 2510B, Conductivity, "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.

16.10. SW-846 Update III, Method 9040B Rev.2, Jan. 1995.

16.11. Method 4500-H+, pH Value, "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.

16.12. Method 9050A, "Specific Conductance", Test Methods for Evaluating Solid Waste, EPA SW-846 Third Edition, 12/96.

17. METHOD MODIFICATIONS

17.1. Deviations from Source Methods and Rationale

17.1.1. Temperature is not reported with the pH measurement. All measurements are made at room temperature (approximately 22°C). The instrument performs automatic temperature compensation.

17.1.2. pH is analyzed as a single analysis.

17.1.3. The instrument guidelines specify switching to 0.1N sulfuric acid for titration at >1500 mg $CaCO_3$/L

17.1.4. The conductivity cell constant is calculated automatically by the equipment, and is not calculated manually by the analyst.

17.1.5. All standards and reagents used for these methods are purchased as certified stock solutions.

18. ATTACHMENTS

18.1. Appendix 1 – Buret Syringe Check

19. REVISION HISTORY

19.1. WS-WC-0028, Revision 4.4, Effective 03/28/2014

19.1.1. Included references to SM 2510B for conductivity.

19.1.2. Appended Section 1.1 - If the autotitrator is malfunctioning or in the case of short hold aqueous samples, manual pH measurement can be performed following procedure in SOP WS-WC-0044.

19.1.3. Inserted Section(s) 10.6 – 10.8 in body of SOP: (19.1.3.1 – 19.1.3.3 below)

19.1.3.1. Burette accuracy is verified annually. Acceptance criteria is %CV is less than or equal to 0.2%. (CV = standard deviation of 10 measurements/average of 10 measurements.) See Table 1 for an example. File is also stored on the public drive under Genchem/AT2 Burette Syringe Calibration.

19.1.3.2. The sample dosing pump accuracy is verified annually. However, based on analysts discretion, this accuracy may be verified more frequently if the analyst suspects that the dosing pump is in need of adjustment based on data evaluation. To determine the pump flow rate, select the PUMP CAL icon in the PC-Titrate software. It is located in the lower left corner of the display screen. The software automatically prompts the analyst once the icon is selected. Follow the instructions. In order to enter the flow rate into the software program, follow this path: Interface → Hardware Set-Up → Digital/Amplifiers. Enter the flow rate that was determined into the dosing meter pump cell. This flow rate is used by the software to calculate the amount of time required to deliver 25ml of sample to the mixing chamber.

19.1.3.3. All pH calculations are temperature compensated. In order to view this in the software, follow this path: Setup → Analysis Schedule → Load → Conductivity/pH → Click on the second "Assign UDV" in the schedule table → Click on EDIT icon. This will show that the Thermistor is checked for temperature compensation. The Thermistor is verified annually

19.1.4. Attached Table 1.- Buret Syringe check

19.1.5. Editorial revisions

19.2. WS-WC-0028, Revision 4.3, Effective 07/20/2012

19.2.1. Section 9.4: Clarified LCS/LCSD requirements.

19.2.2. Inserted Section 10.5: Bracketing of samples with pH calibration points.

19.2.3. Editorial revisions.

19.3. WS-WC-0028, Revision 4.2, Effective 04/15/2011

19.3.1. Added requirement that fresh aliquots of standard be used for instrument calibration in sections 7.41 and 7.42.

19.3.2. Removed all Sections referring to fluoride analysis as the method is not supported by the laboratory.

19.3.3. Editorial corrections.

19.4. WS-WC-0028, Revision 4.1, Effective 03/05/2010

19.4.1. Inserted Section 7.1: Reagent water is produced by a Millipore Nanopure system. Water shall not contain particles larger than 0.20 micron and have a resistance of at least 18 Mohms. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks, in accordance with SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water.

19.4.2. Section 10.2 NOTE: Inserted NOTE: Refer to the following sections in the instrument manufacturer's handbook for additional information. Sections 13.10.5 (pH and alkalinity), 13.12.5 (conductivity), and 13.14.5 (fluoride).

19.5. WS-WC-0028, Revision 4, Effective 1/30/2009

19.5.1. Updated to TestAmerica format.

Appendix 1
Example Buret Syringe Check

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Buret Syringe Check

Buret Syringe Lot#: M06-C1102 **Part#** PC-100-1009
Serial# 019775

Tested by: JCB/LW

Date: January 16,2014

Dispense	container weight (g)	1/10 dispense+conatainer (g	neat weight (g)
1st 1/10	13.3088	15.7954	2.4866
2nd/1/10	13.3597	15.8458	2.4861
3rd 1/10	13.394	15.8878	2.4938
4th 1/10	13.3935	15.888	2.4945
5th 1/10	13.3171	15.817	2.4999
6th 1/10	13.2823	15.7764	2.4941
7th 1/10	13.2762	15.7721	2.4959
8th 1/10	13.4123	15.9127	2.5004
9th 1/10	13.3211	15.8204	2.4993
10th 1/10	13.3686	15.8652	2.4966

Standard Deviation:	0.00502	
Average:	2.49472	
% CV:	0.2011	Pass

Acceptance Criteria: %CV ≤0.2

Buret Syringe Check_011614.xlsData Entry
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Buret Syringe Check v1
1/16/2014 JB

Title: Determination of Chemical Oxygen Demand [Method EPA 410.4]

Approvals (Signature/Date):

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1. SCOPE AND APPLICATION

1.1. This method covers the determination of chemical oxygen demand (COD) in ground and surface waters, domestic and industrial wastes. This SOP is based on EPA 600/Method 410.4, Revision 2, August 1993.

1.2. The applicable range is 0 - 150 mg/L. The acceptable range (0 - 150 mg/L) tests are USEPA approved for NPDES reporting. Higher concentrations can be determined by dilution. Analytical time is approximately 4 hours for a batch of 20 samples.

2. SUMMARY OF METHOD

2.1. Organic matter is oxidized by dichromate in sulfuric acid, which converts most of the carbon to carbon dioxide, and hydrogen to water, according to the following equation (using potassium acid phthalate (KHP), as an example):

$$2\ KC_8H_5O_4 + 10\ K_2Cr_2O_7 + 41\ H_2SO_4 \longrightarrow$$
$$16\ CO_2 + 46\ H_2O + 10\ Cr_2(SO_4)_3 + 11\ K_2SO_4$$

Dichromate ions (Cr2O7-2) form orange-colored solutions. The COD concentration increases as the concentration of hexavalent chromium, (as found in the dichromate ion), decreases. This COD procedure measures hexavalent chromium, which results in a calibration line with a negative slope.

2.2. Samples, blanks and standards in sealed tubes are heated in a Hach block digestor in the presence of dichromate at 150°C. After two hours, the tubes are removed from the digestor, cooled and measured spectrophotometrically at 420 nm.

2.3. Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Low levels of organic matter contamination in water or on glassware may yield biased results. Volatile materials may be lost during sampling and storage. Samples should

completely fill an amber glass bottle and be preserved to pH < 2 with concentrated sulfuric acid. Chlorides are quantitatively oxidized by dichromate and will give a positive interference. Mercuric sulfate is already present in the reagent vials, and will prevent interferences of up to 2000 ppm chloride. However, the chloride concentration below which no positive bias was observed was much lower than advertised for each supplier. If chloride is higher than 1000 ppm, dilutions must be made to reduce this interference.

4.2. Chloride is routinely present in a wide variety of environmental samples and is a common interference in determination of COD. Chloride has both positive and negative impacts on the reagent chemistry under the aggressive oxidizing conditions of COD digestion. It can degrade the performance of the silver catalyst, it can react with organic compounds in the sample and alter their COD response, and it can directly consume the oxidizing agent. While these chemical interactions can generate both positive and negative potential bias in COD results, empirical data indicates that chloride typically generates a positive COD response and high bias in analysis of field samples. Although chloride generally yields a positive bias, the complex and sample-dependent reagent interactions can also generate highly erratic COD results when chloride is present.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The addition of sample material to the reagent tube may create mercury vapor. All samples must be poured inside a fume hood. Reagent tubes may not be opened after analysis except in a fume hood.

5.1.2. COD vials contain sulfuric acid, as well as salts of silver, mercury and dichromate. Return used tubes to original containers for proper disposal.

5.1.3. When mixing samples in vials, the vials will get very hot. Hold the vial by the cap to avoid burns and possibly dropping/breaking the vial.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents

are being handled. Nitrile, latex and vinyl gloves all provide adequate protection against the materials used in this SOP.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
COD (Hach or Environmental Express) Digestion Solution (Digestion Solution for COD)	Caustic Carcinogen Cumulative poison	1 mg/m^3-TWA (Sulfuric Acid) 0.1 mg/m^3-TWA (Mercury)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms of redness, pain, and severe burn can occur to skin and eyes. This product contains high levels of Mercury, Silver and Chromium in Sulfuric Acid.
Mercuric Oxide, Red	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. **May affect the central nervous system.** Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 mg/m^3 TW`A as CrO_3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Potassium Permanganate	Oxidizer	6 mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing and shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness and blurred vision and can cause severe damage possibly permanent.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Hach block digester for 16 mm tubes set at 150 ± 2 ° C (Hach DRB 200)

6.2. Spectrophotometer to measure 16 mm tubes at 420 nm. (ThermoGenesys 20)

6.3. Eppendorf adjustable pipet (10µL - 10 mL) and pipet tips

6.4. Four 100 mL volumetric flasks and one 200 mL volumetric flasks

6.5. Disposable test tubes and caps

6.6. pH test strips

7. REAGENTS AND STANDARDS

7.1. Environmental Express for COD tubes # B1010 (0 - 150 ppm low range)

7.2. Hach Quantab Chloride Test Strips Cat# 27513-40.

7.3. Reagent water -- distilled or deionized water free of the analytes of interest. (ASTM Type 2 or equivalent)

7.4. 10,000 ppm COD stock Calibration standard. A certified 10,000 ppm stock standard obtained commercially, and given a one year expiration from date first opened, or manufacturers expiration date, whichever is less.

7.5. 1,000 ppm COD stock Reference standard. A certified 1,000 ppm stock standard obtained commercially, and given a one year expiration from date first opened, or manufacturers expiration date, whichever is less.

7.6. COD working standards. Dilute stock solutions to final volume (using Class A glassware and/or pipets).

7.7. Reagent grade Potassium Permanganate ($KMnO_4$)

7.7.1. Potassium Permanganate Stock Solution: Dissolve 0.16g of Potassium permanganate with reagent water in a 100 mL volumetric flask. Dilute to volume with reagent water.

7.7.2. Potassium Permanganate, 0.10 mM: Pipet 100 mL of potassium permanganate stock solution into a 100 ml volumetric flask. Dilute to volume with reagent water.

COD Curve QC

Cal Std/ MS/MSD (ppm)	Volume of 10,000 ppm Stock Standard (mL)	Final Volume (mL)
0	0	10
10	0.01	10
50 –CCV	0.05	10
100	0.1	10
150	0.15	10
MS/SD - 50	0.05	10

Ref Std (ppm)	Volume of 1,000 ppm Stock Standard (mL)	Final Volume (mL)
ICV/LCS – 80	0.8	10

Working standards are to be made daily.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in an amber glass or plastic bottles. All bottles must be thoroughly cleansed and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2. Samples must be preserved with concentrated sulfuric acid to a pH less than 2 and cooled to 4°C at the time of collection.

8.3. Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples (maintained at 4˚C) may be held for up to 28 days.

9. QUALITY CONTROL

9.1. The wavelength accuracy of non-scanning (manually advanced) monochromators in VIS/UV-VIS spectrophotometers must be checked annually. The checks must be documented and available for inspection. Any repairs or problems are documented in the maintenance logbook for the spectrophotometer.

9.2. One method blank must be analyzed with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory reagent water processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit. Re-extraction of the blank, other batch QC, and the affected samples is required when the method blank is deemed unacceptable. See WS-PQA-003, Quality Control Policy, for specific acceptance criteria.

9.2.1. The reporting limit for COD is 10 mg/L.

9.3. A Laboratory Control Sample (LCS) must be run with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte are outside control limits. Re-extraction of the blank, other batch QC and all associated samples is required if the LCS is deemed unacceptable. See WS-PQA-003, Quality Control Policy, for specific acceptance criteria.

9.3.1. The LCS recovery must fall within 90-110% recovery.

9.4. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be analyzed with every ten (10) field samples. MS/MSD samples are aliquots of a selected field

sample spiked with analytes of known identity and concentration. The MS/MSD must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided in LIMS or by the client. Re-extraction of the blank, an LCS, the selected field sample, and the MS/MSD may be required after evaluation and review.

9.5. A duplicate control sample (LCSD or DCS) may be substituted when insufficient sample volume is provided to process an MS/MSD pair as required by program or client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 Quality Control Policy, for specific acceptance criteria.

9.5.1. Relative Percent Difference (RPD) of the DCS must be less than 20%.

9.6. The ICV is reported as the LCS and the ICB is reported as the Method Blank. Use the 50 mg/L standard from the curve as the CCV. Use the reference standard as the ICV.

9.7. ICV, CCV and LCS recovery should fall within 90%-110% of the true value.

Acceptance Criteria and Corrective Actions-COD		
QC Type	**Acceptance Criteria**	**Corrective Action**
Calibration Curve-daily 0.0 ppm, 10 ppm, 50 ppm, 100 ppm, 150 ppm	r ≥ 0.995	Reanalyze once. If the problem persists, reprepare the standards, and recalibrate.
ICV/REF/LCS 80 ppm, (Second Source Standard)	90%-110% Recovery, 20% RPD if DCS is analyzed	Reanalyze once. If the problem persists, reprepare the standards, reanalyze, and/or recalibrate.
ICB/CCB/Method Blank	<Reporting Limit	Reanalyze once. If the problem persists, isolate the source of the problem and fix it. If the problem is isolated to the blank, reprepare, reanalyze and proceed. If the problem may have affected previous sample results (i.e. instrument failure, contaminated test tubes, etc.) reanalyze samples bracketed by the failed blank.
CCV (50 mg/L)	90%-110% Recovery	Reanalyze once. If the problem persists, isolate the source of the problem and fix it. If the problem is isolated to the standard (i.e. misspike, scratched test tube, etc.), reprepare, reanalyze and proceed. If the problem may have affected previous sample results (i.e. instrument failure, contaminated test tubes, etc.) reanalyze samples bracketed by the failed standard.
MS/MSD Spike Level = 50ppm	75%-125% Recovery, 20%RPD	If %RPD is in control, and recoveries are outside limits, report data and narrate. If %RPD fails, reprepare MS/MSD once and reanalyze.

10. CALIBRATION

10.1. Annual Spectrophotometer Wavelength Check - The wavelength accuracy of non-scanning (manually advanced) monochromators in VIS/UV-VIS spectrophotometers is checked annually. The wavelengths of two-peak maxima of a dilute solution of Potassium Permanganate (0.10mM KMnO4) are determined on the VIS/UV-VIS spectrophotometer and these determined wavelengths are compared to the true values. (reference Section 16.4 and Section 16.5).

10.1.1. Allow the spectrophotometer to warm up for at least ½ hour before using.

10.1.2. Set the monochromator to a wavelength of 516nm and zero the instrument with reagent water. Fill a test tube with 0.10 mM $KMnO_4$ solution, insert into the instrument and record the absorbance.

10.1.3. Advance the monochromator by a wavelength increment of 5nm, zero the instrument with reagent water, and record the absorbance of the 0.10 mM $KMnO_4$ solution. Continue advancing the monochromator in 5nm increments to 556nm, zeroing, and recording the absorbance of the 0.10 mM $KMnO_4$ solution at each wavelength.

10.1.4. Find and record the instruments wavelengths of maximum absorbance from the tabulation of absorbance values. One maximum absorbance should occur at 526mm and a second at either 541mm or 546mm. If these criteria are not met, the instrument should be serviced by qualified personnel.

10.2. Linear Dynamic Range Verification (LDR) – The linear dynamic range must be verified every six months for each analyte wavelength used on each instrument. The linear dynamic range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear dynamic range limit must be analyzed during a routine analytical run. For the initial determination and semi-annual verification of the upper limit of the LDR for each wavelength, determine the signal response from a minimum of three different concentration standards across the estimated range. One standard should be at or near the upper limit of the estimated range. The concentration measured at the upper limit of the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

10.3. Prepare standards for digestion with the samples per Sections 11.3 - 11.8, using the same reagent tubes as for the samples.

10.4. Samples and standards should be at room temperature before digestion.

10.5. Pipette 2 mL of standard into a pre-made reagent vial.

10.5.1. 2 mL of Nanopure water are used for the 0 point of the curve, ICB/CCBs, and dilutions.

10.5.2. Curve standards - 2 mL of the following standards are added to vials: 0 ppm, 10 ppm, 50 ppm, 100 ppm, 150 ppm. The 50 ppm standard is used for CCV.

10.5.2.1. Curve ICV/REF/LCS is referenced in Section 7.6

10.6. Analysis of standards is per Section 11.9-11.11.
Analyze curve standards in order of decreasing concentration.

10.6.1. Set absorbance to 420 nm. Adjust the instrument to 0 using the 150 ppm standard.

10.6.2. Read other standards on the curve and calculate r value.

10.6.3. The calibration curve must yield a correlation coefficient of 0.995 or better.

10.7. An ICV/REF/LCS and ICB/MB should be analyzed immediately following the calibration. CCV/CCBs should be analyzed after every 10 samples and at the close of the run. ICV/CCV results must be within 10% of the true value. ICB/CCB results must be less than the reporting limit. If a standard fails to meet this criterion, reanalyze the standard once. If reanalysis indicates that the calibration is in control, proceed with analysis. If the reanalysis confirms the calibration is out of control, the source of the problem must be identified and, if necessary, the instrument must be recalibrated. All samples bracketed by failing CCV/CCB must be reanalyzed. See Acceptance Criteria and Corrective Actions – COD table for additional information.

11. PROCEDURE

11.1. Procedural Variations

11.1.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

11.1.2. Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Place block digester in hood and start COD pre-heat until the temperature stabilizes at 150°C.

11.3. Samples and standards should be at room temperature before analysis. Invert samples to homogenize and check pH for proper preservation. Sample pH should be <2. Include the lot number of the pH test strips in the "View Batch Information" tab in TALS and indicate on the "worksheet" tab what the measured pH is.

11.4. Measure the chloride concentration of each sample using the chloride test strips. Record the concentration in the "Worksheet" tab of the TALS batch and include the lot number of the test strips in the "View Batch Information" tab in TALS. If the concentration is > 1,000 mg/L make appropriate dilutions to bring the concentration below 1,000 mg/L. Make sure to include the dilution factor in the "dilution factor" tab in TALS.

11.5. Pipette 2 mL of blank, sample or diluted portion thereof into a pre-made reagent vial. Dilutions, if necessary, should be made prior to adding sample to vial. If sample concentration exceeds the low level curve, switch to the high level curve. Sample dilutions should only be made on the high curve.

11.6. Cap tubes tightly and shake.

WARNING: Tubes will be extremely hot! Hold the tubes by the caps, not the glass.

11.7. Place tubes on 150°C block digester for two hours.

11.8. Remove, mix, and allow tubes to cool. Allow precipitate to settle.

11.9. Allow spectrophotometer to warm up for at least 30 minutes prior to use.

11.10. Test tubes can be inserted directly into spectrophotometer for analysis. Cuvettes are not necessary. Prior to insertion, tubes should be at room temperature, wiped clean with a kimwipe, and inspected for scratches. If scratches are present, the tube should be inserted into the spectrophotometer in such a way that the light does not pass through the scratches.

11.11. Example Sample Analysis Batch

Analysis #	Sample Description
1	ICV/REF/LCS – 2nd source standard
2	ICB/MB
3	Sample 1
4	Matrix Spike (MS)
5	Matrix Spike Duplicate (MSD)
6 - 12	Samples 2 – 8
13	CCV
14	CCB
15 – 24	Samples 9 – 18
25	CCV
26	CCB
27 – 28	Samples 19 – 20
29	CCV
30	CCB

11.12. Routine and Preventive Maintenance

Routine and Preventive Maintenance for Genesys Spectrophotometer
As needed: Replace lamp. Replace fuse.
Annually: Check instrument manual. Perform wavelength calibration. (Section 10.1) Replace lamp annually or when erratic response is observed.

12. CALCULATIONS/DATA REDUCTION

12.1. Create a 410.4_Prep batch in the TALS system. In the “Reagents” tab, enter the appropriate volumes for each standard used (i.e., curve amounts, ICV/CCV amounts, and MS/MSD amounts).

12.1.1. In the “Worksheet” tab, enter the correct initial volumes for each sample. The initial volume should be 10 mL for calibration standards, MS/MSD samples, ICV, LCS, CCVs and CCBs. Initial volumes for field samples should be 2 mL.

12.1.2. Label each calibration point as an “IC”.

12.1.3. Enter the correct final volumes for each sample. The final volume should be

10 mL for calibration standards, MS/MSD samples, ICV, LCS, CCVs and CCBs. Final volumes for field samples should be 2 mL.

12.2. Before creating the 410.4 analytical batch, make sure that the 410.4_Prep batch has been second level reviewed in TALS.

12.3. Create an analytical batch using method 410.4 in the method list in TALS. When prompted to create a batch from scratch or a batch from an existing batch, select the "From Existing Batch" option and enter the batch number obtained in Section 12.1. Be sure to select the "Copy Output Containers" and the "Copy Data Type Info" fields.

12.3.1. In the Batch Editor screen it is imperative to enter "Gen20" in the equipment field. If this is not done the batch will not calculate any results.

12.3.2. In the "Sample Quants" tab, select the Instrument Method from the drop down menu for all samples and QC in the batch. If this is not done the batch will not calculate.

12.3.3. In the "Batch Editor" screen, enter all necessary reagent information, lot number information, and digestion time/temperature data.

12.3.4. In the "Worksheet" tab, enter all initial and final volumes. (See section 12.1.1 and 12.1.3 for these volumes).

12.3.5. In the "Reagent" tab enter the volumes used for the curve. TALs does not need volumes entered for reagents for the LCS/MS/MSD samples. These volumes transferred from the prep batch.

12.3.6. In the "Batch Results" tab enter the response values from the spectrophotometer in the Response field for the calibration samples. After all response values are entered for all calibration standards, right click, select Calibration, select New, and select all calibration points. Click OK and evaluate the linear curve to ensure that the r value is greater than (or equal to) 0.995. If the calibration is acceptable, proceed to Section 12.3.7.

12.3.7. Enter all response values for all remaining samples in the batch. Once all response values have been entered, evaluate the batch to ensure that all QC meet acceptance criteria and all samples fall within the calibration range of the curve. If any samples fall outside of the calibration curve, prepare a High Level curve, and repeat all steps beginning with Section 12.1.

12.3.8. If all QC criteria are within control limits and all other data is correct, set the batch to "1st Level Review" and submit for second level review.

12.3.9. Report only those values that fall between the lowest and highest calibration

standards. Samples exceeding the highest standard should be diluted and reanalyzed.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL is verified via an MDL check every 6 months. Refer to SOP WS-QA-0006 (MDLs and IDLs) for details. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be 1-3 times the MDL.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

14.2. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.3. Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in this method. This will produce less waste for disposal.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Disposable glass COD digestion tubes contaminated with Mercuric Sulfate, Chromate and Sulfuric acid. Once cool and no longer needed, these are returned to the original shipping container. When the container is full, it is moved to the main waste area for disposal as mercury waste. (HACH EZ Recycle COD container may be used for HACH tubes. Part number 2895405P. For Environmental Express tubes, use Heritage Environmental recycle package.)

16. REFERENCES/CROSS REFERENCES

16.1. Methods for the Determination of Inorganic Substances in Environmental Samples, Method 410.4 Revision 2.0, Determination of Chemical Oxygen Demand by Semi-Automated Colorimetry, (August 1993).

16.2. The procedure follows the “Hach COD Closed Reflux Micro method” which closely follows Standard Methods 5220C. The Hach method uses prematured, ready-to-use reagent vials.

16.3. Standard Methods 5220C 18th Edition, 1992, 19th Edition, 1995, 20th Edition, 1998.

16.4. Standard Methods 5220D 20th Edition, 1998.

16.5. Handbook for Analytical Quality Control in Water and Wastewater Laboratories; EPA-600/4, 79-019, U.S. EPA Office of Research and Development: Cincinnati: March, 1979.

16.6. Environmental Laboratory Approval Program Certification Manual; New York DOY; Albany, NY.

16.7. Oxygen Demand, Chemical (USEPA Reactor Digestion Method, Method 8000), DOC316.53.01099, Hach Company, April 2013 (Edition 8)

17. METHOD MODIFICATIONS

17.1. Deviations from reference method.

17.1.1. Commercially available Environmental Express vials are used instead of the recommended digestion/catalyst solution. The following modifications have been made based on the recommendation of the COD vial manufacturer:

17.1.1.1. 2 ml of sample, standard and blank are used instead of 2.5 ml during the digestion procedure. This is necessary to ensure that there is sufficient solution to properly digest the sample/standard/blank.

17.1.1.2. The applicable range listed in method 410.4 is 3 mg/L to 900 mg/L, and has been modified. Low range vials are accurate at low levels and are used to read COD up to 150 mg/L.

17.1.2. Manual colorimetric method is used for COD determination instead of the semi-automated colorimetry procedures. The chemical principles and reactions are the same for both procedures.

17.1.3. For the curve, the spectrophotometer is zeroed using the 150 mg/L standard instead of the 0 mg/L standard. This prevents negative readings on the spectrophotometer. This method measures the amount of Cr6+ that remains following digestion. Because of this chemistry, the absorbance at 420 nm (near the absorbance maximum of 426 nm for Cr6+) is used, rather than 600 nm as referenced in Method 410.4.

17.2. Clarification: Method 410.4 recommends that MDLs should be performed semiannually. TestAmerica Sacramento's policy is to perform MDLs annually, with semiannual verification.

18. ATTACHMENTS

None

19. REVISION HISTORY

19.1. WS-WC-0040, Revision 4.1, Effective 01/23/2015

19.1.1. Section 9.7 – Updated Acceptance Criteria and Corrective Actions – COD table.

19.1.2. Inserted Section 12.3.9 - Report only those values that fall between the lowest and highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

19.1.3. Editorial changes.

19.2. WS-WC-0040, Revision 4.0, Effective 2/10/2014

19.2.1. Updated the SOP with respect to Corporate Quality Policy Memorandum CA-Q-QM-010. This includes removing all reference to a "high range" and "low range" calibration and analysis, instead using a single calibration range and sample dilution to accomplish the same aim. In addition, screening the samples for chloride ion and diluting if necessary based on that result is required.

19.2.2. Updated to prefer use of reagents from Environmental Express rather than from Hach.

19.3. WS-WC-0040, Revision 3.4, Effective 12/31/2013

19.3.1. Section 13.2, inserted following the first sentence, "The MDL is verified via an MDL check every 6 months. Refer to SOP WS-QA-0006 (MDLs and IDLs) for details."

19.3.2. Section 17.1.3, amended to read, "For the low curve, the spectrophotometer is zeroed using the 150 mg/L standard instead of the 0 mg/L standard. This prevents negative readings on the spectrophotometer. This method measures the amount of Cr6+ that remains following digestion. Because of this chemistry, the absorbance at 420 nm (near the absorbance maximum of 426 nm for Cr6+) is used, rather than 600 nm as referenced in Method 410.4.

19.3.3. Added Section 17.1.4, "For the high curve, the amount of Cr3+ which remains following digestion is measured. Because of this chemistry, the absorbance at 620 nm is used, rather than 600 nm as referenced in Method 410.4."

19.3.4. Added Section 17.2, "Clarification: Method 410.4 recommends that MDLs should be performed semiannually. TestAmerica Sacramento's policy is to perform MDLs annually, with semiannual verification.

19.3.5. Added Section 11.13, Routine and Preventive Maintenance Table.

19.4. WS-WC-0040, Revision 3.3, Effective 09/30/2013

19.4.1. Modified Section 7.3 to: "Reagent water -- distilled or deionized water free of the analytes of interest. (ASTM Type 2 or equivalent)."

19.4.2. Rewrote Section 12 to reflect the process in place as a result of the TALS conversion.

19.4.3. Deleted attachment 1. No longer used.

19.4.4. Editorial changes.

19.5. WS-WC-0040, Revision 3.2, Effective 5/25/12.

19.5.1. Updated annual spectrophotometer wavelength check procedures in Section 10.

19.5.2. Editorial changes.

19.6. WS-WC-0040, Revision 3.1, Effective 4/22/2011

19.6.1. Inserted Section 6.5: Disposable test tubes and caps.

19.6.2. Inserted Section 6.6: pH test strips.

19.6.3. Modified Section 11.2 to: Place block digester in hood and start COD pre-heat until the temperature stabilizes at 150° C.

19.6.4. Editorial changes.

Title: EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY GC-FID [SW-846 8015B; IOWA OA-2]

Approvals (Signature/Date):

Dennis Konopka — 5/30/14 — Date GC Department Lead	Michael Ridenhower — 5/30/14 — Date Health & Safety Manager / Coordinator
Tony L. Byrd — 5-30-14 — Date Quality Assurance Specialist	Elaine Wild — 6/2/14 — Date Laboratory Director

This SOP was previously identified as SOP No. ST-GC-0005 Rev. 18

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1.0 SCOPE AND APPLICATION

1.1 This SOP documents the procedure used to determine extractable petroleum hydrocarbons (EPH), also known as total recoverable petroleum hydrocarbons (TRPH), or TPH in environmental samples by Gas Chromatograph with a Flame Ionization Detector (GC/FID).

1.2 Matrices to which this procedure is applicable are water, water miscible wastes, soil, sludges, oil and miscellaneous solids.

1.3 This procedure is amenable to the determination of common hydrocarbon mixtures which are soluble in methylene chloride, are amenable to gas chromatography, and have boiling points between approximately 150 and 450 °C.

1.4 Carbon ranges from C10 to C40 are detected. Typical hydrocarbons are Diesel, Motor Oil, Kerosene, Jet fuel, Mineral Spirits and Hydraulic Oil.

1.5 Sample preparation techniques are described in SOP ST-OP-0002.

1.6 This SOP is based on SW-846 methods 8000C and 8015B, and the IOWA method OA-2.

1.7 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMs).

1.7.1 Additional compounds may be amenable to this procedure. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument. When an MDL study can not be conducted, the MDL is set equal to the reporting limit.

2.0 SUMMARY OF METHOD

2.1 Organic compounds are extracted from neutral pH environmental samples with methylene chloride. The volume of methylene chloride is concentrated to give an extract of known volume.

2.2 A portion of the extract is analyzed by GC/FID. The GC must be previously calibrated using multi-level calibration standards. The petroleum hydrocarbons are quantified as Diesel Range Organics (DRO) or Oil Range Organics (ORO) unless a specific hydrocarbon product is requested by the client.

3.0 DEFINITIONS

3.1 See the Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 For method 8015B:

3.2.1 Diesel is defined as hydrocarbon peaks eluting in the carbon range C10-C28.

3.2.2 Motor oil is defined as hydrocarbon peaks eluting in the carbon range C28-C40.

3.3 For method OA-2, hydrocarbons are determined by their elution pattern.

4.0 INTERFERENCES

4.1 Any GC chromatographable compound, organic or inorganic, which gives a flame detector response in the retention time window selected, will be included in the quantification of total petroleum hydrocarbons. The method is non-specific and all co-extracted compounds are detected.

4.2 Solvents, reagents, glassware and other sample processing hardware may yield artifacts and interferences to sample extracts. Strict attention to glassware cleaning and handling and demonstration of solvent purity will lead to minimization of these interferences.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.2 There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time weighted average			
STEL – Short term exposure limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/FID system: The lab utilizes an Agilent 6890 with dual flame ionization detector (FID) system with autosampler.

6.1.1 The GC column type and instrument run conditions are posted in the front of the maintenance calendars and reside in the Chemstation method.

6.2 Data System – Chemstation for acquisition and Chrom for data processing.

6.3 Amber vials. Crimp top seals.

6.4 Pipettes, disposable

6.5 Micro syringes: 10µL, 250µL, 500µL, 1000µL. Hamilton 1700 Series.

6.6 Analytical Balance, capable of weighing ± 0.01 grams.

6.7 Volumetric flasks, Class A, various volumes

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision. "Recipes" for standards are found in the reagent module of LIMS.

7.2 Primary standards

- 7.2.1 Standards are either purchased from a commercial vendor or prepared by dilution of neat liquid mixtures in methylene chloride.
- 7.2.2 Storage: The standards are stored at -20 to 0° C in amber glass vials with Teflon-lined caps.
- 7.2.3 Shelf life:
  - 7.2.3.1 Unopened ampoules: manufacturer's expiration date.
  - 7.2.3.2 Opened ampoules and Intermediate standards: Manufacturer's expiration date, or 1 year, whichever is sooner.
  - 7.2.3.3 Working standards: Primary standard's expiration date or 6 months, whichever is sooner.
- 7.2.4 Degradation:
  - 7.2.4.1 If standard degradation is indicated, the standard is discarded, irrespective of its' shelf life.

7.3 o-Terphenyl surrogate

7.4 Calibration Standards

- 7.4.1 Calibration standards for total petroleum hydrocarbon analysis may vary according to the requests of clients. A diesel #2 standard and motor oil (a mid-weight oil) are used for initial calibrations. Other hydrocarbons, such as Kerosene, Jet Fuel A, Mineral Spirits, and Fuel Oil #6 can be analyzed upon request.
- 7.4.2 Secondary Standards - Secondary standards for calibration are prepared by dilution in methylene chloride of the primary standards mentioned. The secondary standards are subject to the same storage requirements as the primary standards.
- 7.4.3 Working Calibration Standards - The working calibration standards are prepared in methylene chloride: diesel and motor oil at a minimum of five concentration levels. One of the concentration levels should be ≤ to the concentration of the reporting limit, and the remaining levels should define the expected range of concentrations in the samples or the linear range of the instrument. All working calibration standards expire after 6 months or at the expiration date of their primary standards, whichever is shorter. Working standards are stored in the freezer.
- 7.4.4 Second Source for calibration verification (ICV). Purchased from a different vendor than the calibration standards.

7.5 Carbon range retention time marker standard

- 7.5.1 This marker standard includes C10 (decane), C12 (dodecane), C24 (tetracosane), C28 (octacosane) and C40 (tetracontane).

7.6 Gases for carrier and make- up: Hydrogen carrier, Nitrogen make-up

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Water samples are unpreserved and refrigerated at 4 ± 2 °C.

8.3 Soil samples are refrigerated at 4 ± 2 °C.

8.4 The extraction holding time for water samples is 7 days.

8.5 The extraction holding time for soil samples is 14 days.

8.6 The extract analysis holding time is 40 days from the date of extraction.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 DI water is used as the blank matrix for water batches.

9.2.4 Sodium sulfate is used as the blank matrix for solid batches.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 DI water, spiked with the analytes of interest is used as the LCS for water batches.

9.3.4 Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 **Procedural Variations/ Nonconformance and Corrective Action**

9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 External standard calibration is used.

10.2 Marker Standard

10.2.1 Analyze a carbon range retention time marker standard daily.

10.2.2 This marker standard includes C10 (decane), C12 (dodecane), C24 (tetracosane), C28 (octacosane) and C40 (tetracontane) . Additional markers may be analyzed upon client request.

10.3 Initial Calibration

10.3.1 Inject an aliquot of each level of the calibration standard, minimum 5 points, for the target compounds being analyzed (i.e., diesel and/or motor oil). The low level standard should be at or below the reporting limit. The highest level standard defines the working range of the detector. Quadratic (Second order curves) require 6 calibration points.

10.3.1.1 If additional hydrocarbons are requested (e.g. kerosene), a minimum 5 point calibration for SW 8015 or a minimum 3 point calibration for OA-2 is performed, with the low level standard at or below the reporting limit.

10.3.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., FID replacement).

10.3.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica corporate policy, "Selection of Calibration Points", CA-T-P-002.

10.3.4 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq$ 20%.

10.3.5 If the %RSD in the initial calibration is > 20%, then linear regression may be used to evaluate the curve or recalibration may be done.

10.3.5.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than the reporting limit for the analyte.

10.3.5.2 If a linear regression curve is used, r^2 must be $\geq$ 0.990

10.3.5.3 Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve.

10.4 Initial Calibration Verification (ICV)

10.4.1 The initial calibration verification standard should be from a different standard source than the one used for the initial calibration.

10.4.2 An ICV for the target compounds being analyzed (diesel and/or motor oil) must be performed with every initial calibration.

10.4.2.1 If additional hydrocarbons were requested, an ICV is analyzed for those hydrocarbons.

10.4.3 The ICV performance must be within +/- 20% D criteria for each analyte.

10.4.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard.

10.4.3.2 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.4.3.3 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the

laboratory director. Any such action must be documented in an NCM.

10.5 Continuing Calibration Verification (CCV)

10.5.1 A CCV may be from the same source as the calibration or a second source.

10.5.2 Analyze a CCV for each target hydrocarbon being analyzed. It is not necessary to analyze a CCV for every hydrocarbon having an initial calibration, **only** those being analyzed.

10.5.3 **8015 requirements**

10.5.3.1 Analyte response factors must be verified at the beginning of each analytical run, (by either an ICV or a CCV), after every 10 samples and at the end of the analytical run through the analysis of a mid-level calibration standard.

10.5.3.2 A CCV %D must be within +/- 20% D for each analyte.

10.5.4 **OA-2 requirements**

10.5.4.1 Analyte response factors must be verified on each working day by the measurement of one or more calibration standards. The laboratory runs a calibration standard at the beginning of each analytical run, (by either an ICV or a CCV), after every 10 samples and at the end of the analytical run through the analysis of a mid-level calibration standard.

10.5.4.2 The ending CCV may be omitted for OA-2.

10.5.4.3 A CCV %D must be within +/- 20% D for each analyte.

10.5.5 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument.

10.5.5.1 If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed.

10.5.5.2 If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require calibration and all samples run after the last passing CCV must be reanalyzed.

10.6 Retention Time Windows

10.6.1 Retention Time Windows: analytes and surrogate(s):

10.6.1.1 Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

10.6.1.2 The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the midpoint standard of the initial calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

10.6.1.3 If the retention time window as calculated above is less than +/- 0.015 minutes, use +/- 0.015 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

10.6.1.4 A new retention time window study is performed annually or when the analytical column from a new vendor or different stationary phase is used. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

10.6.2 Retention Time Window for Target Analytes

10.6.2.1 Retention Time windows for hydrocarbons are established daily. The retention time window is based on the retention time of the applicable carbon marker standard.

10.6.3 Corrective Action for Retention Times

10.6.3.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

11.0 PROCEDURE

11.1 Allow standards, samples and sample extracts to reach ambient temperature before analysis.

11.2 All analysis conditions and injection volumes for samples must be the same as for the calibration standards.

11.3 Sample introduction:

11.3.1 The samples are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.4 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them at -20 to 0°C and protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 External Standard Calculations

12.2.1 Analyte Concentration (µg/L) in sample

Concentration (µg/L):

$$[C] = \frac{A_x * V_t * D}{\overline{CF} * V_i * V_s}$$

Where:

$[C]$	=	*Analyte Concentration in sample (µg/L)*
A_x	=	*Area of peak (response)*
V_t	=	*Total volume of extract (µL)*
D	=	*Dilution factor*
$\overline{CF}$	=	*Calibration factor ($\overline{RF}$ in target) – Response factor*
V_i	=	*Volume of extract injected (µL)*
V_s	=	*Volume of sample extracted*

12.2.2 *On column concentration (ug/mL):*

$$[OC] = \frac{A_x}{\overline{CF}}$$

Where:

$[OC]$ = *On Column Concentration [typically expressed in µg/mL (ppm)]*

Then substitute/derive

$$[C] = [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

When *on column concentration* $[OC]$ is equal to the *CAL-AMT (calibration amount)* of the low level standard needed to support the *reporting limit (µg/L)* and we solve the equation for *concentration (µg/L)*

Then

$$[C] \equiv RL \equiv [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

Where:

RL = Reporting Limit

12.3 Manual Integrations

12.3.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica policy: CA-Q-S-002, Manual Integration. Manual integrations are denoted with a "M" flag on the Target quantitation report.

12.4 Use the total integrated area that results from a common baseline (include all of the area down to the baseline) between the retention time limits established, to quantify total extractable petroleum hydrocarbons in the sample.

12.5 Quantitation of Hydrocarbons

12.5.1 Total Area Response is the total response in the chosen retention time window.

12.6 Dilutions

12.6.1 If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range.

12.6.2 It may be necessary to dilute samples due to matrix.

12.7 Carryover

12.7.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.7.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.7.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 OA-2 evaluation:

13.1.1 For hydrocarbons, the analyst should rely primarily on pattern recognition.

13.1.2 Retention time windows are used for identification, but the "pattern" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the pattern resembles the standard chromatogram.

13.1.3 Weathering can make pattern determination difficult. Often the early eluting peaks of the hydrocarbon are diminished and peaks may not be in the anticipated ratios. If severe weathering is indicated, the analyst may base hydrocarbon pattern on the general shape of the chromatography (e.g. diesel crown) and later eluting peaks.

13.2 8015 evaluation:

13.2.1 For 8015, evaluation is based on carbon ranges, not pattern. All peaks observed within the prescribed carbon range are used in quantitation.

13.2.2 If the peaks observed appear as a single peak (e.g. PAHs) rather than "hydrocarbon like" (multi-peaks), the analyst may exclude them from quantitation. An NCM is written to document the reason for the exclusion.

13.3 Weathering, single peaks and comments regarding pattern should be documented using the NCM process.

13.4 The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.5 Method Blank

13.5.1 Acceptance Criteria:

13.5.1.1 No target analytes may be present in the method blank above the reporting limit.

13.5.1.2 The method blank must have acceptable surrogate recoveries.

13.5.1.3 Corrective Action for Method Blanks not meeting acceptance criteria:

13.5.1.3.1 <u>Method Blank Contamination</u> – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.5.1.3.2 <u>Method Blank Surrogate excursion</u> – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.6 Laboratory Control Sample (LCS)

13.6.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.

13.6.1.1 The LCS must have acceptable surrogate recoveries. .

13.6.1.2 Corrective Action for LCS not meeting acceptance criteria:

13.6.1.2.1 <u>LCS Spike Recovery excursion (high)</u> – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. . In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

13.6.1.2.2 <u>LCS Spike Recovery excursion (low)</u> – batch is re-prepped and re-analyzed.

13.6.1.2.3 <u>LCS Surrogate Recovery excursion</u> – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the

LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.

13.6.1.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM

13.6.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.6.3 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.

13.6.4 Corrective Action for MS/MSD not meeting acceptance criteria:

13.6.4.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

13.6.5 Sample result evaluation

13.6.5.1 Dilutions

13.6.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.6.5.1.1.1 Dilution: Sample– An NCM is written to document the reason for the dilution.

13.6.5.1.1.2 Dilution: Surrogate(s) and/or spike(s) diluted out– An NCM is written to document the reason for the dilution.

13.6.5.2 Carryover

13.6.5.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.6.5.2.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.6.5.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.6.5.3 Insufficient Sample

13.6.5.3.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. An NCM is written to document the insufficient volume.

14.0 [redacted]

14.1 Method performance data, reporting limits, and QC acceptance limits, are maintained in the LIMs.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 [redacted]

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 Waste Streams Produced by the Method

16.1.1 The following waste streams are produced when this method is carried out.

16.1.1.1 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type “D”.

16.1.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 SW-846 ,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods”, Method 8000C

17.2 SW-846 ,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods” Method 8015B.

17.3 IOWA Method OA-2 University Hygienic Laboratory, Iowa City, IA, rev. 07/27/93.

17.4 St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.5 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.6 TestAmerica Policy CA-Q-S-002, Manual Integration

17.7 TestAmerica Policy CA-T-P-002, Selection of Calibration Points

17.8 Associated SOPs

17.8.1 ST-OP-0001, Labware Preparation for Organic Analysis

17.8.2 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series

17.8.3 ST-PM-0002, Sample Receipt and Chain of Custody

17.8.4 ST-QA-0002, Standard and Reagent Preparation

17.8.5 ST-QA-0005, “Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.”

17.8.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.8.7 ST-QA-0016, IDL/MDL Determination
17.8.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 MODIFICATIONS FROM REFERENCE METHOD

18.1 Chapter 1 of SW-846 states that the Method Blank should not contain any analyte of interest at or above the Method Detection Limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

19.0 CHANGES TO PREVIOUS SOP REVISION

19.1 No Changes, Annual Review.
19.2 Rev 16;
- 19.2.1 Annual Review, No Changes

19.3 Rev 17:
- 19.3.1 Updated scope in section 1.0.
- 19.3.2 Removed Gasoline Range Organics (GRO) from the section 2.0.
- 19.3.3 Removed the definition for gasoline for method 8015B in section 3.0.
- 19.3.4 Updated equipments and supplies throughout section 6.0.
- 19.3.5 Updated calibration standards in sections 7.4, 7.5 and 7.6.
- 19.3.6 Updated the retention time standard being analyzed daily in section 10.2.
- 19.3.7 Updated initial calibration in section 10.3.
- 19.3.8 Updated ICV and CCV information in sections 10.4 and 10.5.
- 19.3.9 Updated the storage temperature for extracts in section 11.0.
- 19.3.10 Added external calibration calculations to Section 12.2
- 19.3.11 Updated the quantitation of hydrocarbons in section 12.5.
- 19.3.12 Removed the reference to “Quantims” and “Clouseau”.
- 19.3.13 Updated corrective action for non-conformances in section 13.0.
- 19.3.14 Corrected references to Corporate Policies in Sections 17.6 & 17.7.

19.4 Rev 18:
- 19.4.1 Added carbon range retentiontime marker to Section 7
- 19.4.2 Changed “stock” to “primary” for clarification in Section 7
- 19.4.3 Added six point requirement for quadratic curves to Section 10.
- 19.4.4 Added requirement for NCM when excluding peaks to Section 13.
- 19.4.5 Added location of Control Limits (found in LIMS) to Section 13.

19.5 Rev 19
- 19.5.1 Updated equipment and supplies in Section 6
- 19.5.2 Added names of marker standards in section 7.5.1 and 10.2.2
- 19.5.3 Corrected fragmented sentences in section 10.3.5
- 19.5.4 Added retention time windows criteria in section 10.6.1.4

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Title: AROMATIC VOLATILES AND VOLATILE PETROLEUM HYDROCARBONS BY GC-PID/FID [SW-846 8015B, IOWA OA-1]

Approvals (Signature/Date):

Dennis Konopka — Date 5/29/14 GC Lab Lead	Michael Ridenhower — Date 5/29/14 Health & Safety Manager / Coordinator
Tony L. Byrd — Date 5-30-14 Quality Assurance Specialist	Elaine Wild — Date 6/2/14 Laboratory Director

This SOP was previously identified as SOP No. ST-GC-0014 Rev 11

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes sample preparation and analysis of volatile petroleum hydrocarbons, Gasoline, by a purge and trap GC-PID/FID procedure, following method OA-1. .

1.2 This SOP describes sample preparation and analysis of volatile petroleum hydrocarbons. Gasoline Range Organics (GRO) correspond to the range of alkanes from C6 to C10 and cover a boiling point range of approximately 60°C-170°C. This procedure follows SW-846 methods 8000C and 8015B.

1.3 Water samples and soil samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of the analytes in soil may be determined by a methanol extraction procedure.

1.4 Water samples are prepared in accordance with SW-846 method 5030A. Soil samples are prepared in accordance with SW-846 method 5035. For specific client projects, the superceded SW-846 method 5030 may be used for soil analyses.

1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Helium gas is bubbled through the sample heated at 40°C, and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a column where the volatile components are absorbed. After purging is completed, the column is heated and flushed with inert gas to desorb the components onto a gas chromatographic column. Hydrogen gas carries the analytes onto the GC column, where they are separated and finally detected by a Photo Ionization Detector (PID) and Flame Ionization Detector (FID) in tandem.

2.1.1 The GC is hooked up to two detectors that work in tandem. Analytes leave the column and enter the PID. Compounds vent from the PID and go into the FID for analysis by EPA Method 8015B and Iowa OA-1 Method

2.2 If an aqueous or solid sample exhibits high level contamination, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. An aliquot is extracted with methanol and combined with water in a purging chamber, and analyzed by purge-and-trap GC following the standard procedure for water samples.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 For the Method 8015B, Gasoline is defined as hydrocarbon peaks eluting in the carbon range C6-C10.

3.3 For the OA-1 Method, Gasoline is defined by its elution pattern.

4.0 INTERFERENCES

4.1 Impurities in the purge gas, and organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared

from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. The determination of carryover is mad by checking for trap contamination, and examining the positive hits in each sample against those is the sample run just before it.

4.4 The method is non-specific and all co-extracted compounds will be detected. Any GC chromatographable compound, organic or inorganic, which gives a flame detector response in the retention time window selected will be included in the quantification of total petroleum hydrocarbons.

5.0 [illegible]

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them. There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, the analyst need to either turn off the power to the instrument, or disconnect it from its source of power.

5.3 PRIMARY MATERIALS USED
The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a de-fatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weight Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph (GC), Agilent 5890 with a Flame Ionization Detector (FID) system with Archon autosampler, purge and trap concentrator.

6.2 GC column type, trap, and instrument run conditions are posted on the individual GC instruments.

6.3 Micro syringes – 10-µL, 25-µL, 100–µL, 250–µL, 500-µL, and 1000-µL. These should be equipped with a 20 gauge (0.006” ID) needle.

6.4 Gas tight syringes – 5 mL and 25 mL.

6.5 Volumetric flasks, Class A

6.6 pH paper

6.7 Vortex mixer

6.8 Analytical Balance, capable of weighing ± 0.01 grams

6.9 Glass beads: Store in a drying oven.

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.

7.2 Methanol: purge and trap grade.

7.3 Water: HPLC grade or equivalent.

7.4 Surrogate Spike Mix: a,a,a-Trifluorotoluene in methanol.
- 7.4.1 This standard has a maximum lifetime of six months.

7.5 Calibration standards, NIST traceable:
- 7.5.1 The Gasoline standard in methanol at a concentration of 1000 ug/mL.
- 7.5.2 Working standards are good for 6 months from the date made, unless the parent expires first. If component breakdown occurs replace the standard.
- 7.5.3 Gases for carrier and make- up: Hydrogen- carrier, Air – makeup.

7.6 ICV standards, NIST traceable:
- 7.6.1 Gasoline in methanol at a concentration 2500-µg/mL.
  - 7.6.1.1 Due to manufacturer differences in hydrocarbon types, the ICV is from the same vendor but a separate standard lot from the calibration standard.

7.7 Marker Standards 2-methylpentane and 1,2,4-trimethylbenzene

7.8 The aromatic volatiles ICV standard is a second source from the calibration standard.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Aromatic volatiles water samples are preserved with 1:1 HCl and stored at 4 ± 2 °C. Analysis hold time is 14 days from collection.

8.3 GRO water samples may or may not be chemically preserved with HCl and stored at 4 ± 2 °C. Analysis hold time is 14 days from collection with or without chemical preservation.

8.4 Aqueous samples are stored in 40mL VOA vials with Teflon lined septa at $4^{o}C \pm 2^{o}C$, with minimum headspace.

8.5 Soil samples are refrigerated at 4 ± 2 °C. Analysis hold time is 14 days from collection.

8.5.1 Medium level solid extracts are aliquoted into40 mL VOA vials with Teflon lined caps and stored at $4^{o}C \pm 2^{o}C$. The extracts are stored with minimum headspace.

8.6 For 5035 analysis

8.6.1 Solid samples, for low level analysis, may be field preserved with sodium bisulfate solution, or collected unpreserved using the Encore (or equivalent)™ sampler and shipped to the laboratory within 48 hours of sampling.

8.6.1.1 Following shipment back to the lab the soil is preserved with sodium bisulfate or kept frozen until analysis.

8.6.1.2 It is recommended that two Encore (or equivalent) samplers be used for each field sample position, to allow for any reruns than may be necessary.

8.6.2 Solid samples, for medium level analysis, may be field extracted with methanol, or collected unpreserved using the Encore (or equivalent)™ sampler and shipped to the laboratory within 48 hours of sampling.

8.6.2.1 It is recommended that two Encore (or equivalent) samplers be used for each field sample position, to allow for any reruns than may be necessary.

8.6.2.2 Solid samples – field extracted with methanol

8.6.2.2.1 Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2.2.2 Seal the bottle and attach a label.

8.6.2.2.3 Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.2.2.4 Ship with appropriate sampling instructions.

8.6.2.2.4.1 At client request, the methanol addition and weighing may also be performed in the field.

8.6.2.2.4.2 When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.2.3 Solid samples – field extracted with methanol

8.6.2.3.1 When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a pre-weighed VOA vial containing 5mL methanol (25mL methanol for the 25g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.7 An additional sample is collected for percent moisture determination.

8.8 At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab.

8.8.1 This is an older procedure based on method 5030A.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a <u>method blank</u>, a <u>Laboratory Control Sample</u> (LCS), <u>Matrix Spike</u> (MS) and <u>Matrix Spike Duplicate</u> (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
9.2.2 A method blank must be analyzed with every analysis water or low soil batch.
9.2.3 A method blank must be prepared with every medium level soil batch (20 or fewer samples of the same matrix). The medium level method blank is tied to an extraction and does not require repeated analysis with each sample analysis batch.
9.2.4 For Water analyses, the method blank is comprised of HPLC water.
9.2.5 For Soil analyses, the method blank is comprised of glass beads.

9.3 **Laboratory Control Sample**
9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.3.2 An LCS must be analyzed with every analysis water or low soil batch.
9.3.3 An LCS must be prepared with every medium level soil batch (20 or fewer samples of the same matrix).
9.3.3.1 The medium level LCS is tied to an extraction and does not require repeated analysis with each sample analysis batch.
9.3.4 For Water analyses, the LCS is comprised of HPLC water fortified with gasoline.
9.3.4.1 For water analyses, the LCS and ICV are the identical. If an ICV was already run in the 12 hour analysis clock, the analyst may chose to omit running a second LCS. The ICV is uploaded as the LCS.
9.3.5 For Soil analyses, the LCS is comprised of glass beads fortified with gasoline.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**
9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Surrogate**
9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.
9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**
9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 External standard calibration is used.

10.2 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).

10.3 **FID Initial Calibration**
10.3.1 Prepare a multi-point calibration curve by fortifying 5ml of HPLC water with incrementing concentrations of Gasoline standard.

10.3.2 Analyze standards in ascending order.

10.3.3 The initial calibration contains a minimum of 5 points, for each target. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector, with the highest level standard establishing the linear range of the instrument.

10.3.3.1 Note OA-1 requires a minimum 3 point calibration. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector, with the highest level standard establishing the linear range of the instrument.

10.3.4 For initial calibration criteria see section 10.6

10.4 **Initial Calibration Criteria**

10.4.1 SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.

10.4.2 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.

10.4.3 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.

10.4.4 **Linear calibration using the average response factor**

10.4.4.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq$ 20% for each target analyte.

10.4.4.2 If the %RSDs in the initial calibration is > 20%, then calibration using a linear regression may be employed.

10.4.5 **Linear calibration using a least squares regression**

10.4.5.1 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.

10.4.5.2 r (correlation coefficient) must be $\geq$ 0.995 OR r^2 (coefficient of difference) must be $\geq$ 0.990.

10.4.5.3 **Linear calibration using a least squares regression, forcing thru zero**

10.4.5.3.1 Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression though zero for some calibrations.

10.4.5.3.2 Curve must still meet criteria in 10.6.5.1 and 10.6.5.2.

10.4.5.4 **Linear calibration using a least squares regression, weighting of data points**

10.4.5.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. For this reason it may preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) to improve accuracy at the low end of the curve.

10.4.5.4.2 Curve must still meet criteria in 10.6.5.1 and 10.6.5.2.

10.4.6 **Non-linear calibration**

10.4.6.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed.

10.4.6.2 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.4.6.3 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.

10.4.6.4 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990.

10.4.6.5 If a quadratic curve fit is used, a minimum of 6 points are required.

10.5 **Initial Calibration Verification (ICV)**

10.5.1 The initial calibration verification standard should be from a different standard source than the one used for the initial calibration. Due to manufacturer differences in hydrocarbon types, the Gasoline ICV is from the same vendor but a separate standard lot from the calibration standard.

10.5.2 An ICV must be performed with each initial calibration.

10.5.3 The ICV performance must be within ± 20% D criteria for each analyte.

10.5.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard.

10.5.3.2 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.5.3.3 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.6 **Continuing Calibration Verification (CCV)**

10.6.1 A CCV may be from the same source as the calibration or may be from a second source.

10.6.2 Analyte response factors must be verified at the beginning of each analytical run (either by an ICV or a CCV), after every 10 samples and at the end of the analytical run, by analyzing a mid-level calibration standard.

10.6.2.1 The ending CCV may be omitted for OA-1.

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10.6.3 The gasoline CCV %D must be within ± 15%D.

10.6.3.1 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument.

10.6.3.1.1 If this second CCV meets the acceptance criteria the analysis run may continue; however, the preceding samples must be reanalyzed.

10.6.3.1.2 If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require calibration.

10.7 **Marker Standards**

10.7.1 Marker Standards are run daily per the requirements of the method being utilized.

10.7.1.1 Method 8015 GRO analysis: Marker standards used to determine the retention time range are 2-methylpentane and 1,2,4-trimethylbenzene.

10.8 **Retention Time (RT) Windows**

10.8.1 Retention Time (RT) windows must be determined for all analytes.

10.8.1.1 Establishing RT windows:

10.8.1.1.1 Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used).

10.8.1.1.2 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes,whichever is greater.

10.8.1.1.3 The center of the retention time window is the retention time from the average of three standards used to calculate the RT window.

10.8.1.1.4 The center of the window is updated with the midpoint standard of the initial calibration.

10.8.1.1.5 A new retention time window is established each time a new column is installed or the GC column has been shortened during maintenance.

10.8.1.1.5.1 The new windows must be generated within one week of the installation of the new column.

10.8.1.1.5.2 Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

10.8.2 Retention Time Criteria

10.8.2.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.8.2.1.1 If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

10.8.2.1.2 The retention time range for GRO is defined during initial calibration. Two specific gasoline components are used to establish the range, 2-methylpentane and 1,2,4-trimethylbenzene. The procedure described in Method 8000C is used to establish the retention time windows for these two components. The GRO retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component.

11.0 PROCEDURE

11.1 Allow standards, samples and sample extracts to reach ambient temperature before analysis.

11.2 All analysis conditions and injection volumes for samples must be the same for the calibration standards. (Including purge time and flow desorb time and temperature, column temperatures, multiplier setting etc.).

11.3 Water, soil and medium level extract analyses are all performed by heated purge (40°C).

11.4 **Water Sample Preparation (5030B method)**

11.4.1 Remove 5ml sample with 5ml syringe.

11.4.2 Transfer the 5ml sample into an empty labeled 40ml VOA vial.

11.4.3 Add 10ul of surrogate to sample.

11.4.3.1 Prepare a method blank with 10µL surrogate using 5ml of HPLC water.

11.4.3.2 Prepare a LCS with10µL surrogate and 10µL of spike using 5ml of HPLC water.

11.4.3.3 For samples designated for MS/MSD analysis, add 10µL of spiking solution.

11.4.4 Seal the vial.

11.4.5 Check and document the pH of the sample of the remaining sample.

11.4.5.1 Do not check pH prior to taking aliquot for analysis.

11.5 **Low-Level Soil Preparation (5035A method)**

11.5.1 If samples arrive unpreserved, the laboratory must, within 48 hours of collection, preserve samples with sodium bisulfate or by freezing.

11.5.1.1 Check the preparation method code of the SAC to determine preservation.

11.5.1.1.1 Sodium bisulfate code: 4D

11.5.1.1.2Freezing code: 4P

11.5.2 <u>If samples arrive in the Encore (or equivalent) sampler and require Sodium bisulfate preservation</u>:

11.5.2.1 Pre-weigh a labeled 40ml VOA vial.

11.5.2.1.1Label with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction.

11.5.2.2 Extrude the soil sample from the Encore (or equivalent) sampler into the VOA vial.

11.5.2.3 Weigh the vial to the nearest 0.05g.

11.5.2.4 Record weight on the label and runlog.

11.5.2.5 Add a magnetic stir bar, approximately 1g of sodium bisulfate and 5mL of HPLC water.

11.5.2.5.1Soils containing carbonates may effervesce when adding the sodium bisulfate solution. If this is the case, retrieve a second Encore sample plug, add 5mL of water instead, and freeze at <10°C until analysis.

11.5.2.6 Seal the vial.
11.5.2.7 Add 10µL surrogate through the septum to each sample and QC.
11.5.2.7.1 For samples designated for MS/MSD analysis, add 10µL of spiking solution to the vials.
11.5.2.7.2 Prepare a Method Blank using 5.0g of glass beads and 5mL of HPLC grade water. Add 10µL surrogate to the vial.
11.5.2.7.3 Prepare a LCS using 5.0g glass beads, and 5ml HPLC water. Add 10µL surrogate (8015B) and 10µL of spiking solution.

11.5.3 <u>If samples arrive 40ml VOA vial already preserved with Sodium bisulfate</u>:
11.5.3.1 Add 10µL surrogate through the septum to each sample and QC.
11.5.3.1.1 For samples designated for MS/MSD analysis, add 10µL of spiking solution to the vials.
11.5.3.1.2 Prepare a Method Blank using 5.0g of glass beads and 5mL of HPLC grade water. Add 10µL surrogate to the vial.
11.5.3.1.3 Prepare a LCS using 5.0g glass beads, and 5ml HPLC water. Add 10µL surrogate and 10µL of spiking solution.

11.5.4 <u>If samples arrive in the Encore (or equivalent) sampler and require freezing as preservation:</u>
11.5.4.1 Freeze sample until time of analysis.
11.5.4.2 Pre-weigh a labeled 40ml VOA vial.
11.5.4.2.1 Label with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction.
11.5.4.3 Extrude the soil sample from the Encore (or equivalent) sampler into the VOA vial.
11.5.4.4 Weigh the vial to the nearest 0.05g
11.5.4.5 Record weight on the label and runlog.
11.5.4.6 Add a magnetic stir bar, and 5mL of HPLC water.
11.5.4.7 Add 10µL surrogate to each sample and QC.
11.5.4.7.1 For samples designated for MS/MSD analysis, add 10µL of spiking solution to the vials.
11.5.4.7.2 Prepare a Method Blank using 5.0g of glass beads and 5mL of HPLC grade water. Add 10µL surrogate to the vial.
11.5.4.7.3 Prepare a LCS using 5.0g glass beads, and 5ml HPLC water. Add 10µL surrogate and 10µL of spiking solution
11.5.4.8 Seal the vial.

11.5.5 <u>If samples arrive in 40ml VOA vial and require freezing as preservation</u>:
11.5.5.1 Freeze sample until time of analysis.
11.5.5.2 Add 5mL of HPLC water
11.5.5.3 Add 10µL surrogate to each sample and QC
11.5.5.3.1 For samples designated for MS/MSD analysis, add 10µL of spiking solution to the vials.
11.5.5.3.2 Prepare a Method Blank using 5.0g of glass beads and 5mL of HPLC grade water. Add 10µL surrogate to the vial.
11.5.5.3.3 Prepare a LCS using 5.0g glass beads, and 5ml HPLC water. Add 10µL surrogate and 10µL of spiking solution of spiking solution.
11.5.5.4 Seal the vial.

11.6 **<u>Low-Level Soil Preparation (superseded 5030A method)</u>**
11.6.1 See SOP ST-QA-0038 for the procedure for sub sampling.
11.6.2 Weigh 5g ± 0.5 of the sample into a pre-weighed 40mL glass labeled vial.
11.6.2.1 If the sample is suspected or known to have high concentrations of analytes, reduce the sample aliquot to 1.0 g
11.6.3 Record the weight.
11.6.4 Add 5ml HPLC water
11.6.5 Add 10µL surrogate
11.6.6 Seal the vial.
11.6.7 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.6.7.1 Prepare a Method Blank using 5.0g of glass beads and 5mL of HPLC grade water, add 10µL surrogate to the vial.
11.6.7.2 Prepare a LCS using 5.0g glass beads, 5µL of surrogate and 20µL of spiking solution.
11.6.7.3 For samples designated as for MS/MSD, add 10µL of spiking solution of spiking solution.

11.7 **Methanol Extraction of Soils (5035A method)**
11.7.1 Extrude the (nominal) 5g sample into a pre-weighed VOA vial containing 5mL methanol (25mL methanol for the 25g sampler).
11.7.2 Obtain the weight of the soil added to the vial and note on the label.
11.7.2.1 Prepare a Method Blank using 5.0g of glass beads and 10mL methanol. Add 8µL surrogate parent solution
11.7.2.2 Prepare a LCS using 5.0g glass beads, 10mL methanol, 8µL surrogate parent solution and 5µL medium level spiking mix (8015B).
11.7.3 Using a vortex mixer, agitate sample for at least half a minute.

11.8 **Methanol Extraction of Soils (superseded 5030A method)**
11.8.1 See SOP ST-QA-0038 for the procedure for sub sampling.
11.8.2 Weigh 5 g ± 0.5 of the sample into a pre-weighed 40mL glass labeled vial.
11.8.2.1 If the sample is suspected or known to have high concentrations of analytes, reduce the sample aliquot to 1.0 g
11.8.3 Record the weight.
11.8.4 Add 10mL of purge and trap methanol.
11.8.4.1 Prepare a Method Blank using 5.0g of glass beads and 10mL methanol. Add 8µL surrogate parent solution to the vial.
11.8.4.2 Prepare a LCS using 5.0g glass beads, 10mL methanol, 8µL surrogate parent solution and 5µL medium level spiking solution .
11.8.4.3 For the MS/MSD, add 8ul surrogate and 5ul medium level spiking mix medium level spike mix.
11.8.5 Seal the vial.
11.8.6 Using a vortex mixer, agitate sample for at least half a minute.

11.9 **Volatile Analysis**:
11.9.1 Load each 40ml VOA sample vial (and QC) in the purge and trap autosampler.
11.9.1.1 Medium Level Analysis
11.9.1.1.1Fill gas-tight syringe with 5ml HPLC water.
11.9.1.1.2 Add sample methanol extract to the syringe (no more than 100µL for a 5mL purge).
11.9.1.1.2.1 If less than 1µL of methanol extract is to be added to the water, dilute the methanol extract using a serial dilution.
11.9.1.1.3Transfer methanol extract/HPLC water in syringe to a labeled 40ml VOA vial.
11.9.2 Record autosampler sample analysis sequence in logbook.
11.9.3 Start analysis.
11.9.4 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition.
11.9.5 After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.
11.9.6 When the standards and extracts are not being used, refrigerate them at 4 $\pm$ 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 External Standard Calculations

12.2.1 See Target software for calculations.

12.3 Manual Integrations

12.3.1 Identified compounds are reviewed for proper integration. Integrations are performed automatically by the data system. If necessary, manual integrations are performed and are documented by the analyst. Manual integrations are denoted with a "M" flag on the Target quantitation report. See Corporate policy: S-Q-004, Manual Integration.

12.4 Quantitation of Hydrocarbons

12.4.1 Quantitate any peaks within the retention times from n-hexane to n-decane (excluding the surrogate) as gasoline using the average calibration factor from the initial calibration.

12.4.2 Use the total integrated area that results from a common baseline (include all of the area down to the baseline) between the established retention time limits, to quantify total volatile petroleum hydrocarbons in the sample.

12.5 Dilutions

12.5.1 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.

12.5.2 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.

12.5.2.1 Aqueous samples requiring less than a 1:5000 dilution can be diluted directly using a 5-ml syringe

12.5.2.2 Low level soil samples may re-analyzed using a 1g sample aliquot or utilizing the methanol extraction technique.

12.6 Carryover

12.6.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.6.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.6.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, carryover may have occurred.

12.6.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred

12.6.1.3 If the chromatographic profile resembles the previous sample, the results are questionable.

12.6.1.3.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Method OA-1 evaluation:

13.1.1 For hydrocarbons, the analyst should rely primarily on pattern recognition.

13.1.2 Also used for identification are Retention Time windows in tandem with the "pattern" produced by major peaks of these analytes in the standard. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the pattern resembles the standard chromatogram.

13.1.3 Weathering can make pattern determination difficult. In weathering, the early eluting peaks of the hydrocarbon are often diminished and peaks may not be in the anticipated ratios. If severe weathering is indicated, the analyst may base hydrocarbon pattern on the general shape of the chromatography (e.g. diesel crown) and later eluting peaks.

13.1.4 Weathering, single peaks and comments regarding pattern, issues or anomalies should be documented using the NCM process.

13.2 Method 8015B evaluation:

13.2.1 For 8015B, evaluation is based on carbon ranges, not pattern. All peaks observed within the prescribed carbon range are used in quantitation.

13.2.2 If the peaks observed, appear single peak in nature (e.g. BTEX) rather than "hydrocarbon like" (multi-peaks), the analyst may exclude them from quantitation.

13.2.3 Weathering, single peaks and comments regarding pattern, issues or anomalies should be documented using the NCM process.

13.3 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036.

13.4 Method Blank

13.4.1 Acceptance Criteria:

13.4.1.1 No target analytes may be present in the method blank above the reporting limit.

13.4.1.2 The method blank must have acceptable surrogate recoveries.

13.4.1.3 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet.

13.4.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.4.2.1 Method Blank Contamination – Blank contamination above the RL (>½ RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.4.2.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.5 Laboratory Control Sample (LCS)

13.5.1 Acceptance Criteria:

13.5.1.1 All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.5.1.2 The LCS should have acceptable surrogate recoveries.

13.5.2 Corrective Action for LCS not meeting acceptance criteria:

13.5.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

13.5.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.5.2.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.

13.5.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM

13.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.6.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.6.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.6.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

13.7 Surrogate

13.7.1 All Surrogates should be within established control limits for accuracy (%Recovery).

13.7.2 Corrective Action for Surrogate not meeting acceptance criteria:

13.7.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.8 Sample result evaluation

13.8.1 Dilutions

13.8.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.8.1.1.1 Dilution: Sample – An NCM is written to document the reason for the dilution.

13.8.1.1.2 Dilution: Surrogates(s) and/or Spike(s) diluted out – Dilution: Surrogate(s) and/or spike(s) diluted out – An NCM is written to document the reason for the dilution

13.8.2 Carryover

13.8.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.8.2.2 If a sample is analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.8.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.8.2.3.1 The carryover affected analyses are not reported, unless specifically requested by the client or there was insufficient extract/sample remaining to reanalyze.

13.8.3 Insufficient Sample

13.8.3.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. An NCM is written to document the insufficient volume.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS..

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

16.2 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to ST-HS-0001, "Waste Minimization Plan".

16.3 Waste Streams Produced by the Method

16.3.1 The following waste streams are produced when this method is carried out.

16.3.1.1 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".

16.3.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 8000C

17.2 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" Method 8015B

17.3 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", (Sample Prep- Purge and-Trap), Method 5030A and 5030B.

17.4 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", (Sample Prep- Purge and-Trap), Method 5035.

17.5 IOWA Method OA-1University Hygienic Laboratory, Iowa City, IA, rev. 07/27/93.

17.6 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.7 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.8 TestAmerica Policy CA-Q-S-001, Acceptable Manual Integration Practices

17.9 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

17.10 Associated SOPs

17.10.1 ST-OP-0001, Labware Preparation for Organic Analysis
17.10.2 ST-PM-0002, Sample Receipt and Chain of Custody
17.10.3 ST-QA-0002, Standard and Reagent Preparation
17.10.4 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
17.10.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.10.6 ST-QA-0016, IDL/MDL Determination
17.10.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.10.8 ST-QA-0038, Procedure for sub sampling.

18.0 MODIFICATIONS FROM REFERENCED METHOD

18.1 Chapter 1 of SW-846 states that the Method Blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank, following consultation with the client.

18.2 Method OA-1 only requires a 3 point calibration. We routinely perform a 5 point calibration

19.0 changes to previous SOP revision

19.1 Rev 9: No changes made annual review.
19.2 Rev 10:
- 19.2.1 Marker standards add to Sections 7 and 10
- 19.2.2 Defined retention time for GRO in Section 10.10

19.3 Rev 11:
- 19.3.1 Updated calibration standards for gasoline in methanol in section 7.5.
- 19.3.2 Updated surrogate and spiking amounts for methods 8015B and 8021B throughout section 11.0.

19.4 Rev 12:
- 19.4.1 Remove 8021B references throughout SOP.
- 19.4.2 Remove spiking amounts for 8021B throughout SOP
- 19.4.3 Section 13 updated with corrective actions

Title: PCB GC ANALYSIS [SW-846 8000B/8000C/8082A; EPA 608]

Approvals (Signature/Date):

11/17/14 Joseph Hunt — Date PCB Analyst	11/17/14 Michael Ridenhower — Date Health & Safety Manager / Coordinator
11-17-14 Marti Ward — Date Quality Assurance Manager	11/17/14 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-GC-0015 Rev. 14

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes procedures to be used for the analysis of polychlorinated biphenyls (PCB) by GC/ECD. The PCBs are determined and quantitated as multi-component Aroclor mixes.

1.2 Sample preparation techniques are described in SOP ST-OP-0002.

1.3 This SOP is based on EPA SW-846 Methods 8000B, 8000C and 8082A, and EPA Method 608.

1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

1.4.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

2.1 Aqueous samples are prepared for analysis using separatory funnel liquid / liquid extraction or continous liquid/liquid extraction techniques. Solid samples are prepared using sonication. Wipes are extracted by autoshaker.

2.2 After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. The external standardization procedure is used.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Interferences in GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.2 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). For PCBs the most common cleanup procedure is the Sulfuric Acid cleanup (Method 3665A).

4.3 Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, DDD) may interfere. When suspected, a standard of DDT analogs should be injected to determine which of the aroclor peaks may be subject to said interference, [DDT may interfere with the last major Aroclor 1254 peak in soil/sediment samples.]

4.4 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or high biased results.

4.5 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample extracts. Strict attention to glassware cleaning and handling and demonstration of solvent purity will lead to minimization of these interferences.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
Hexane	Flammable Irritant	500 ppm-(TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limits refer to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/ECD system: The lab utilizes a Hewlett Packard GC 5890 dual ECD system and an Agilent GC 6890 dual micro ECD system with autosampler.

6.1.1 Columns used Restek Rtx-CLPesticides 30 meter, 0.53 mmID, 0.5µm df; Restek Rtx-CLPesticides2 30 meter, 0.52 mmID, 0.42µm df

6.1.2 GC column types, and instrument run conditions are posted in the maintenance calendar and reside in the Chemstation method.

6.2 Data System – Chemstation for acquisition and Chrom™ for data processing.

6.3 Amber and/or clear glass vials. Crimp top seals.

6.4 Disposal pipettes.

6.5 Micro syringes – 10-µL, 250-µL, 500-µL, 1000-µL. Hamilton 1700 series.

6.6 Volumetric flasks, Class A

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 See recipes for standards in the TALS Reagent module. Information listed in appendix to this SOP.

7.3 PCB primary standard solutions:

7.2.1 Primary standards are prepared by dilution of neat liquid Aroclor mix 1016/1260, and from single aroclor mixes in hexane. Primary standards must be replaced after 6 months or the manufacturer's expiration date whichever is shorter. Standards must be stored in refrigerator or freezer at ≤ 6˚C.

7.3 Working standards:

7.3.1 The working standards are prepared in hexane from the primary standard solution for a minimum of five concentration levels of the Aroclor mix 1016/1260 and one level of the single aroclors. Working standards must be replaced after 6 months or manufacturer's expiration date whichever is shorter. All working standards expire after six months or at the expiration date of their stock standards, whichever comes sooner.

7.4 Gases for carrier and make-up: Hydrogen carrier, Nitrogen make-up.

7.5 Decachlorobiphenyl (surrogate)

7.6 Copper powder

7.6.1 Remove oxides (if powder is dark) by treating with dilute nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

7.7 DDT analog standard – used when possible interference from DDT analogs is present. This is a single standard containing DDT, DDE and DDD at a concentration judged sufficient by the analyst.

7.8 Initial Calibration Verification (ICV) spiking standard is similar to calibration standards, but are from a different source or vendor and are prepared and stored in the same way as calibration standards.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Water samples are unpreserved and stored at 4 ± 2˚ C.

8.3 Soil samples are refrigerated at 4 ± 2˚ C.

8.4 Extracts must be refrigerated at $\leq 6^{o}$C.

8.5 Sample extracts need to be isolated from all potential contaminants and all standards.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples, and/or 24 hours (12 hours for GC/MS).

9.1.3 An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

9.1.4 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.5 Each analytical batch may contain up to 20 environmental samples, a Method Blank (MB), a single Laboratory Control Sample (LCS) and a Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair. In the event that there is insufficient sample to analyze an MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.6 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).

9.2.3 DI water is used as the blank matrix for water batches.

9.2.4 Sodium sulfate is used as the blank matrix for solid batches.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every batch.

9.3.3 DI water, spiked with the analytes of interest is used as the LCS for water batches.

9.3.4 Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches

9.4 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 Additional MS/MSDs do not count towards the 20 samples in an analytical batch.

9.4.3 An MS/MSD can be prepared with every batch, although it is not a method requirement. If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed.

9.5 **Procedural Variations/Nonconformance and Corrective Action**

9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 External standard calibration is used.

10.2 Initial Calibration

10.2.1 Prepare an Aroclor 1016/1260 and Decachlorobiphenyl (surrogate) standard at a minimum of five concentration levels. (Six points are required if a quadratic (second order) curve is used.) The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Table 1. [NOTE: Quadratic regression is not allowed for South Carolina compliance work.}

10.2.1.1 If a specific Aroclor is of interest for a particular project, that Aroclor may be used for the five point calibration rather than the 1016 / 1260 mix. See Client Requirements Sheet or Memo.

10.2.1.2 A single point calibration for Aroclor 1221, 1232, 1242, 1248, 1254, 1262, and 1268 is performed with every initial calibration for pattern recognition.

10.2.1.2.1If any of the above single point Aroclors are detected, samples may be required to be re-analyzed under a five point calibration for the aroclor found. See Client Requirement Sheet to determine if this is needed.

10.2.1.3 Select 3-5 major peaks in the analyte pattern. Calculate the response using the area of these individual peaks.

10.2.1.3.1Aroclor 1221 uses only 3 peaks due to the limited amount of peaks available to choose from.

10.2.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., ECD replacement).

10.2.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Corporate policy, “Calibration Point Selection”, CA-Q-T-002.

10.3 SW 8082 criteria

10.3.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq$ 20%.

10.3.2 If the %RSDs in the initial calibration is > 20%, then calibration using a linear regression may be employed.

10.3.2.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than + or – the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.

10.3.2.2 If a linear regression curve is used, r must be $\geq$ 0.99

10.3.2.3 For South Carolina compliance work, forcing through zero is NOT allowed.

10.3.2.4 Weighting of data points

10.3.2.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it may preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4 608 Criteria

10.4.1 Method 608 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from a curve if necessary to meet 608 calibration criteria. The lowest level of the curve must be at or below the reporting limit. The other standards define the working range of the detector.

10.4.1.1 Refer to the TestAmerica Corporate policy, "Calibration Point Selection", CA-Q-T-002.

10.4.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq$ 10%.

10.4.3 If the %RSDs in the initial calibration is > 10%, then calibration using a linear regression may be employed.

10.4.3.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than + or – the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.

10.4.3.2 If a linear regression curve is used, r must be $\geq$ 0.995

10.4.3.3 Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

10.4.3.3.1 Weighting of data points

10.4.3.3.2 The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.5 Initial Calibration Verification (ICV)

10.5.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.5.1.1 The ICV is not performed for the single point aroclors.

10.5.2 An ICV must be performed with every initial calibration.

10.5.2.1 A passing ICV may be used as the opening CCV for a set of samples run following the ICV.

10.5.3 The ICV performance must be within +/- 20% D criteria.

10.5.3.1 Only the analytes present in the ICAL are evaluated for the 20% criteria.

10.5.3.2 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.5.3.3 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.6 Continuing Calibration Verification (CCV)

10.6.1 A CCV may be a second source or the same source as the initial calibration standards and should be made to represent the midpoint of the curve.

10.6.2 Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a CCV.

10.6.3 It is adequate to verify calibration with a single mixture of Aroclors 1016 and 1260.

10.6.3.1 For projects with specific Aroclor requirements, a specific Aroclor may be included in the daily calibration check.

10.6.4 The calibration verification is acceptable if the %D for both 1016/1260 and the surrogate (DCB) is $\leq$ 20%.

10.6.4.1 The same criterion is used if other Aroclor verifications are performed.

10.6.4.2 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument.

10.6.4.3 If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed.

10.6.4.4 If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).

10.7 Retention Time (RT) Windows

10.7.1 Retention Time (RT) windows must be determined for all analytes.

10.7.2 Establishing RT windows:

10.7.2.1 Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used).

10.7.2.2 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

10.7.2.3 The center of the retention time window is the retention time from the average of three standards used to calculate the RT window.

10.7.2.4 The center of the window is updated with the midpoint standard of the initial calibration.

10.7.2.5 Method 8000B requires a new retention time window study be performed annually or when the analytical column from a new vendor or different stationary phase is used. Method 8000C also requires a new study when a column is clipped during maintenance.

10.7.2.5.1 The new windows must be generated within one week of the installation of the new column.

10.7.2.5.2 Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

10.7.3 Retention Time Criteria

10.7.3.1 The retention times of AR 1016/1260 (and other aroclors if applicable) in each continuing calibration must be within the retention time windows established.

10.8 Method Detection Limit Studies

10.8.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

11.1 Allow standards, samples and sample extracts to reach ambient temperature before analysis.

11.2 Sulfur Removal

11.2.1 Sulfur Removal with Copper Powder

11.2.1.1 Transfer 1.0 mL of sample extract, and associated QC, into labeled vials.
11.2.1.2 Add approximately 2g cleaned copper powder to the vial.
11.2.1.3 Mix for one minute on a mechanical shaker.
11.2.1.4 Allow phases to separate.
11.2.1.5 Separate extract from copper by drawing the extract off with a disposable pipette.
11.2.1.6 Transfer the supernate to a clean, labeled vial.

11.3 All analysis conditions and injection volumes for samples must be the same as for the calibration standards.

11.4 Sample Introduction

11.4.1 Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them at $\leq 6^{o}$C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 External Standard Calculations

12.2.1 *Analyte Concentration (µg/L) in sample*

Concentration (µg/L):

$$[C] = \frac{A_x * V_t * D}{\overline{CF} * V_i * V_s}$$

Where:

$[C]$	=	*Analyte Concentration in sample (µg/L)*
A_x	=	*Area of peak (response)*
V_t	=	*Total volume of extract (µL)*
D	=	*Dilution factor*
$\overline{CF}$	=	*Calibration factor ($\overline{RF}$ in target) – Response factor*
V_i	=	*Volume of extract injected (µL)*
V_s	=	*Volume of sample extracted*

12.2.2 *On column concentration*

On Column Concentration (µg/mL):

$$[OC] = \frac{A_x}{\overline{CF}}$$

Where:

$[OC]$ = *On Column Concentration [typically expressed in µg/mL (ppm)]*

Then substitute/derive

$$[C] = [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

When *on column concentration* $[OC]$ is equal to the *CAL-AMT (calibration amount)* of the low level standard needed to support the *reporting limit (µg/L)* and we solve the equation for *concentration (µg/L)*

Then

$$[C] \equiv RL \equiv [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

Where:

RL = Reporting Limit

12.2.3 See Chrom software for additional calculations.

12.3 Manual Integrations

12.3.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica policy: CA-Q-S-002, "Manual Integrations". Manual integrations are denoted with an "M" flag on the Chrom quantitation report.

12.4 Identification of Aroclors

12.4.1 Tentative identification of an Aroclor occurs when multi-component peaks are found within their respective retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required.

12.4.2 Definitive Aroclor identification is based primarily on pattern recognition. Retention times and retention time windows are used to tentatively identify Aroclors, but the fingerprint produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.4.3 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern. Source-specific information, such as historical data, indicating the anticipation of Aroclors must be documented. The pattern of peaks can serve as confirmation depending of the client specific project requirements.

12.5 Quantitation of Aroclors

12.5.1 Use three to five major peaks when calibrating Aroclors. Choose peaks distinctive of the individual Aroclor. Any manual integration made in the ICAL levels must be noted and be made in any subsequent samples to maintain consistency with the initial calibration. These same three to five peaks are then used to calculate the response/concentration of the Aroclor(s) when present in a sample.

12.5.1.1 For Aroclor 1221 only three peaks are used due to the limited number of peaks available.

12.5.1.2 In instances where less than five peaks are used those peaks that are not used are said to be "dropped" and an NCM must be written. When quantitating Aroclors 1016/1260 in an LCS/D and/or MS/MSD, all five peaks must be used. In samples, less than the standard five peaks may be used to quantitate target analytes if there is demonstrated matrix interferences and/or if multiple, overlapping Aroclors are present. If there is a predominance of one Aroclor that elutes next to and shares peaks with another Aroclor and it is apparent to the analyst that the lesser Aroclor's concentration is elevated significantly by the more dominant Aroclor, then three peaks may be dropped and an NCM written. It is never allowable to quantitate an Aroclor using only one peak.

12.5.2 If well distinguishable Aroclor patterns are present, then multiple Aroclors are quantitated and reported.

12.5.2.1 Aroclor elution times may overlap and one or more Aroclor peaks may be "shared" with another Aroclor. When this occurs, only the predominant Aroclor is quantitated and reported. Aroclors sharing elution time and peaks include: 1016, 1232, 1242, 1248 – these cannot be identified together (unless quantitating an MS/D where Aroclor 1016 is known to be present). Aroclors 1260 and 1262 also share peaks; only one of these can be reported (unless quantitating and MS/D where 1260 is known to be present).

12.5.3 Dual Column Quantitation

12.5.3.1 Dual column confirmation is required for positive Aroclor identification. A secondary column using an alternate phase is employed and the sample is injected simultaneously into both a primary and secondary column. Elution times often differ, as does overall pattern/fingerprint of Aroclors between the two columns. Determination of target analytes on the secondary column is made in the same way as on the primary column. Target analytes may be reported from either column.

12.5.3.2 Method 8000C states to report the lower result of the two columns, unless the Client SOW requires that the higher result be reported. Method 8000B requires the reporting of the higher of the two columns. See Client Requirement Sheet for determination.

12.5.3.2.1 For non-detect (ND) results, report from the primary channel if all QC and CCVs are acceptable. If the QC and CCVs are only acceptable on the secondary column, report the non-detects from this column.

12.5.3.2.2 If the %D between the two columns is greater than 40%, report the higher result if there are obvious chromatographic interferences on the column with the lower result.

12.5.3.2.3 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions.

12.5.3.2.3.1 If there is no evidence of chromatographic problems, report the lower result for method 8000C and the higher result for method 8000B. The data user should be advised of the disparity between the results on the two columns.

12.5.3.2.3.2 Use the higher result if there is obvious chromatographic interference on the column with the lower result.

12.5.3.3 The QC should be reported from the column that reflects the column used for the majority of the samples associated with the QC.

12.5.3.4 The surrogate should be reported from the column used for the reporting of the sample results.

12.6 Dilutions

12.6.1 If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample data is “E” flagged and the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Target analytes with resulting concentrations lower than the dilution adjusted RL should be flagged with a “J” qualifier.

12.6.2 It may be necessary to dilute samples due to matrix.

12.7 Carryover

12.7.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.7.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.7.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (MB)

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet. South Carolina requires blank be below the RL. If blank > RL, take corrective action and re-analyze associated samples.

13.2.1.3 The Method Blank must have acceptable surrogate recoveries.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.2.2.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). . South Carolina requires the LCS to recover within limit of 70 -130%. If recovery is outside limits take corrective action and re-analyze associated samples.

13.3.1.2 The LCS must have acceptable surrogate recoveries.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

13.3.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.3.2.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.

13.3.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM.

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

13.5 Surrogate

13.5.1 All Surrogates should be within established control limits for accuracy (%Recovery).

13.5.2 Corrective Action for Surrogate not meeting acceptance criteria:

13.5.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample Result Evaluation

13.6.1 Dilutions

13.6.2 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.1.1.1 Dilution: Sample– An NCM is written to document the reason for the dilution

13.1.1.2 Dilution: Surrogate(s) and/or spikes diluted out– Dilution: Surrogate(s) and/or spike(s) diluted out– An NCM is written to document the reason for the dilution.

13.6.3 Carryover

13.6.3.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.6.3.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.6.3.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.7 Insufficient Sample

13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report case narrative.

14.0 METHOD [redacted]

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.1 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 [redacted]

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".

16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

16.2.1.4 Expired primary and working PCB standards shall be segregated and placed into the proper satellite accumulation container specifically for PCB waste which is located within the GC lab.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B and 8000C.

17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8082A, 8081B Update IV, February 2007and EPA 608 Method.

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision

17.4 Corporate Environmental Health and Safety Manual (CW-E-M-001), current revision

17.5 TestAmerica Policy CA-Q-S-002, Manual Integrations

17.6 TestAmerica Policy CA-Q-T-002, Calibration Point selection

17.7 Associated SOPs

17.7.1 ST-OP-0001, Labware Preparation for Organic Analysis

17.7.2 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series

17.7.3 ST-OP-0003, Extraction of PCB in Oil

17.7.4 ST-QA-0002, Standard and Reagent Preparation

17.7.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

17.7.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.7.7 ST-QA-0016, IDL/MDL Determination

17.7.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

17.7.9 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 MODIFICATIONS FROM REFERENCE METHOD

18.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

18.1.1 Method Blanks for South Carolina compliance work MUST be below the RL

18.2 The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

18.3 Method 608 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from a curve if necessary to meet 608 calibration criteria. The lowest level of the curve must be at or below the reporting limit.

18.4 SW846 Method 8000C requires that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the sample the laboratory receives, and the number of times the GC column may require clipping, TestAmerica St. Louis does not perform a RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.

19 [redacted]

19.1 Updated the table one regarding the levels of calibration of Aroclor 1016/1260 and the amount of the surrogate used, Decachlorobipenyl.

19.2 Rev 10:

19.2.1 Removing holding times for PCB's in section 8.0 per method

19.3 Revision 11:

19.3.1 Removed references to QuantIMS and Clouseau – replaced with LIMS.

19.3.2 Added software information to Section 6.

19.3.3 Added composition of Method Blank and LCS to Section 9.

19.3.4 Added requirement for 6 points for non-linear curves to Section 10.

19.3.5 Added Reporting limit calculations to Section 12

19.3.6 Added specific corrective actions to Section 13

19.3.7 Updated text in Section 15 to include methods beyond those approved by EPA.

19.4 Rev 12 (4/21/14):

19.4.1 Removed continuous extractions in section 2.

19.4.2 Replaced Target with Chrom data systems in section 6.

19.4.3 Updated location of instrument run setting in section 6.

19.4.4 Added location of standard recipes to section 7.

19.4.5 Updated retention time window study requirements in section 10.

19.4.6 Added exception to SW846 requirements in section 18.

19.4.7 Updated Initial Calibration sequence example following Table 1.

19.5 Revision 13 (9/18/14):

19.5.1 Section 18 updated to reflect requirements for Method Blanks associated with South Carolina compliance work.

19.5.2 Returned continuous liquid/liquid extraction to Section 2.1.

19.6 Revision 14 (10/30/14)

19.6.1 Revision done to combine this SOP with requirements for South Carolina compliance work originally found in SOP ST-GC-0015SC, Rev 1.

19.6.2 Updated Section 10 to include instruction that forcing through zero is NOT allowed for South Carolina compliance work.

19.6.3 Updated Section 10.7 to include requirements from both 8000B and 8000C regarding timing of running retention time window studies.

19.6.4 Added calculations to Section 12.

19.6.5 Updated Section 12.5.3 to include instruction from both 8000B and 8000C in regards to dual column reporting requirements.

19.6.6 Updated Section 13.3 to include South Carolina requirements for LCS recoveries.

19.7 Revision 15 (12/1/14)

19.7.1 Section 4 updated to address possible interference from the DDT analogs

19.7.2 Section 6 updated to include specific columns used.

19.7.3 Section 10 was updated to include a note that quadratic calibrations are not allowed for South Carolina compliance work; MDL study requirements add.

19.7.4 Section 13 was updated to include the 70 – 130% LCS recovery requirement for South Carolina compliance work.

19.7.5 Added standard preparation and concentration information to the appendices.

Table 1								
Calibration Levels ng/ml								
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	
Aroclor 1016/1260	50	100 Reporting Limit	250	500	1000 ICV/CCV	2000	4000	
The surrogate is included with all calibration mixes at the following levels								
Decachlorobiphenyl	2.5	5	12.5	25	50	100	200	

* Level 1 is optional.
Aroclors 1232, 1221, 1242, 1248, 1254, 1262 and 1268 may be quantitated within the range 100 to 4000 ng/mL

Analytical Sequence

Example Initial Calibration

Injection #
1 Conditioning standard
2 Solvent blank
3 Aroclor 1221
4 Aroclor 1232
5 Aroclor 1242
6 Aroclor 1248
7 Aroclor 1262
8 Aroclor 1268
9-15 Aroclor 1254 Levels 1-7
16 Independent Calibration Verification (ICV) standard
17-23 Aroclor 1016/1260 Levels 1-7
24 Independent Calibration Verification (ICV) standard
25-34 Sample Injections (max.10)
35 Aroclor 1016/1260 Level 5

Appendix 1

Standards:

Surrogate Stock – 1000 ug/mL

Aroclors Stock – 1000 ug/mL
(1221, 1232, 1242, 1248, 1254, 1016/1260, 1262, 1268

Intermediates:

Aroclor(s) Intermediate (excluding 1016/1260) – 1 mL Aroclor stock + 50 µl of Surrogate stock to 25 mL with hexane = 40 ug/mL Aroclor/0.2 ug/ml surrogate

Working Standards:
1016/1260 working – 1 ml of 1016/1260 stock + 50 µ of Surrogate stock to 250 ml with hexane = 4 ug/ml 1016.1260/0.2 ug/ml surrogate

Aroclor(s) working – 5 ml of aroclor intermediate to 20 ml with hexane = 4 ug/ml aroclor/0.2 ug/ml surrogate

Calibration:
Aroclor 1016/1260 and 1254

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	80x	0.05
2	40x	0.10
3	20x	0.20
4	8x	0.50
5	4x	1.00
6	2x	2.00
7	1x	4.00
ICV (second source)	4x	1.00

Calibration:
Aroclor 1221, 1232, 1242, 1248, 1262, 1268

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
5	4x	1.00

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8082SurrDeca_00018

Reagent ID: 8082SurrDeca_00018

Type:	ASTD	Expiration Date:	02/28/2015
Description:	Decachlorobiphenyl Solution	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Saulters, Camme N
Storage Location:	Organic Prep Standards Storag	Vendor:	Ultra Scientific
Reagent Volume:	1.000 mL	Vendor Lot #:	CC-3147Z
Creation Date:	08/26/2013	Vendor Cat #:	PPS-150
Container(s):	190362		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
DCB Decachlorobiphenyl (Surr)					1004.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8082 Spike_00018

Reagent ID: 8082 Spike_00018

Type:	ASTD	Expiration Date:	06/01/2019
Description:	8082 spike aroclhor 1060/1260 (PCB)	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Saulters, Camme N
Storage Location:	Organic Prep Standards Storag	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A094177
Creation Date:	08/26/2013	Vendor Cat #:	32039
Container(s):	190372		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1016					1000.00000	ug/mL
PCB-1260					1000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Aroclor1254_00010

Reagent ID: Aroclor 1254_00010

Type:	ASTD	Expiration Date:	08/31/2016
Description:	Aroclor 1254 Solution	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Ultra Scientific
Reagent Volume:	1.000 mL	Vendor Lot #:	CE-2632
Creation Date:	01/15/2013	Vendor Cat #:	PP-352
Container(s):	74708		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1254					100.00000	ug/mL
PCB-1254 Peak 1					100.00000	ug/mL
PCB-1254 Peak 2					100.00000	ug/mL
PCB-1254 Peak 3					100.00000	ug/mL
PCB-1254 Peak 4					100.00000	ug/mL
PCB-1254 Peak 5					100.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ar 1221_00002

Reagent ID: Ar 1221_00002

Type:	ASTD	Expiration Date:	12/31/2018
Description:	Aroclor 1221 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A090667
Creation Date:	07/08/2013	Vendor Cat #:	32007
Container(s):	158536		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1221					1000.00000	ug/mL
PCB-1221 Peak 1					1000.00000	ug/mL
PCB-1221 Peak 2					1000.00000	ug/mL
PCB-1221 Peak 3					1000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ar 1268_00004

Reagent ID: Ar 1268_00004

Type:	ASTD	Expiration Date:	12/31/2019
Description:	Aroclor 1268 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A097795
Creation Date:	06/09/2014	Vendor Cat #:	32410
Container(s):	381748		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1268					1000.00000	ug/mL
PCB-1268 Peak 1					1000.00000	ug/mL
PCB-1268 Peak 2					1000.00000	ug/mL
PCB-1268 Peak 3					1000.00000	ug/mL
PCB-1268 Peak 4					1000.00000	ug/mL
PCB-1268 Peak 5					1000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ar 1242_00002

Reagent ID: Ar 1242_00002

Type:	ASTD	Expiration Date:	11/30/2018
Description:	Aroclor 1242 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A090182
Creation Date:	07/08/2013	Vendor Cat #:	32009
Container(s):	158544		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1242					1000.00000	ug/mL
PCB-1242 Peak 1					1000.00000	ug/mL
PCB-1242 Peak 2					1000.00000	ug/mL
PCB-1242 Peak 3					1000.00000	ug/mL
PCB-1242 Peak 4					1000.00000	ug/mL
PCB-1242 Peak 5					1000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ar 1232_00002

Reagent ID: **Ar 1232_00002**

Type:	ASTD	Expiration Date:	11/30/2018
Description:	Aroclor 1232	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A090290
Creation Date:	07/08/2013	Vendor Cat #:	32008
Container(s):	158538		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1232					1000.00000	ug/mL
PCB-1232 Peak 1					1000.00000	ug/mL
PCB-1232 Peak 2					1000.00000	ug/mL
PCB-1232 Peak 3					1000.00000	ug/mL
PCB-1232 Peak 4					1000.00000	ug/mL
PCB-1232 Peak 5					1000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ar 1262_00002

Reagent ID: Ar 1262_00002

Type:	ASTD	Expiration Date:	06/30/2019
Description:	Aroclor 1262 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A094073
Creation Date:	07/08/2013	Vendor Cat #:	32409
Container(s):	158599		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1262					1000.00000	ug/mL
PCB-1262 Peak 1					1000.00000	ug/mL
PCB-1262 Peak 2					1000.00000	ug/mL
PCB-1262 Peak 3					1000.00000	ug/mL
PCB-1262 Peak 4					1000.00000	ug/mL
PCB-1262 Peak 5					1000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ar 1248_00002

Reagent ID: **Ar 1248_00002**

Type:	ASTD	Expiration Date:	04/30/2019
Description:	Aroclor 1248 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A092864
Creation Date:	07/08/2013	Vendor Cat #:	32010
Container(s):	158550		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1248					1000.00000	ug/mL
PCB-1248 Peak 1					1000.00000	ug/mL
PCB-1248 Peak 2					1000.00000	ug/mL
PCB-1248 Peak 3					1000.00000	ug/mL
PCB-1248 Peak 4					1000.00000	ug/mL
PCB-1248 Peak 5					1000.00000	ug/mL

Title: PESTICIDE GAS CHROMATOGRAPHIC ANALYSIS [SW-846 8081B; EPA 608]

Approvals (Signature/Date):

Charles Hardin — Date 11/17/14 Pesticide Analyst	Michael Ridenhower — Date 11/17/14 Health & Safety Manager / Coordinator
Marti Ward — Date 11-17-14 Quality Assurance Manager	Elaine Wild — Date 11/17/14 Laboratory Director

This SOP was previously identified as SOP No. ST-GC-0016 Rev. 17

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes procedures to be used for the analysis of Pesticides by GC/ECD.

1.2 Sample preparation techniques are described in SOP ST-OP-0002.

1.3 This SOP is based on SW-846 Methods 8000B, 8000C and 8081B and EPA Method 608.

1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

1.4.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

2.1 Aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction. Solid samples are prepared using sonication. Waste dilution and wipes are extracted by autoshaker.

2.2 After the initial preparation step, the sample is introduced to the GC, equipped with capillary columns and dual Electron Capture Detectors (ECD). Concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. The external standardization procedure is used.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.2 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620).

4.3 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results.

4.4 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample extracts. Strict attention to glassware cleaning and handling and demonstration of solvent purity will lead minimization of these interferences.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the

safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2.3 The ECD contains a source of radiation.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/ECD system: The lab utilizes an Agilent GC 6890 dual micro ECD system with auto-sampler.

6.1.1 The GC column type, and instrument run conditions are posted in the maintenance log and reside in the Chemstation method.

6.1.2 Columns used: Restek Rtx-CLPesticides 30 meter, 0.53 mmID, 0.5 µm df and Restek Rtx-CLPesticides2 30 meter, 0.53 mmID, 0.42 µm df

6.2 Data System – Chemstation for acquisition and Chrom for data processing

6.3 Disposable pipettes

6.4 Amber vials: Crimp top seals

6.5 Clear vials and silicon crimp seals

6.6 Micro syringes- 10-µL, 250-µL, 500-µL, 1000-µL. Hamilton 1700 series

6.7 Volumetric flasks, Class A

6.8 Analytical Balance, capable of weighing ± 0.01 g

6.9 Instrument run conditions can be found in the front of the maintenance calendars.

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Pesticide stock standard solutions must be replaced after 6 months, once opened, or manufacturer's expiration date whichever is shorter. Pesticide calibration solutions must be refrigerated at ≤ 6 °C and protected from light. Intermediate and working standards must be replaced at least every six months, or the stock solutions expiration date, whichever is sooner. Additionally standards are discarded if comparison with check standards indicates a problem.

7.3 See reagent log for specific information regarding standards and reagents. See Appendix 1and 2 for examples.

7.4 ICV standards, NIST traceable:
- 7.4.1 The Pesticide ICV standard is a second source from the calibration standard.
- 7.4.2 ICV standard is prepared and stored in the same way as calibration standards.

7.5 Surrogate Standards
- 7.5.1 Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards.

7.6 Column Degradation Evaluation Mix (PEM Standard)
- 7.6.1 A standard containing 4,4'-DDT and Endrin must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after 6 months, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. Refer to Table 1 for details of the column degradation evaluation mix.

7.7 Gases for carrier and make-up: Hydrogen carrier, Nitrogen make-up

8.0 SAMPLE PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Water samples are unpreserved and stored at 4 ± 2 ˚C.

8.3 Soil samples are refrigerated at 4 ± 2 ˚C.

8.4 The extraction holding time for Pesticides analysis in waters is 7 days.

8.5 The extraction holding time for Pesticides analysis in soil/solid matrix is 14 days.

8.6 Extracts must be refrigerated at ≤ 6 °C and analyzed within 40 days of the end of the extraction.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 DI water is used as the blank matrix for water batches.

9.2.4 Sodium sulfate is used as the blank matrix for solid batches.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 DI water, spiked with the analytes of interest is used as the LCS for water batches.

9.3.4 Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 For Method 608 a matrix spike must be performed at a frequency of one per ten samples.

9.5 **Surrogate**

9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.

9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 External standard calibration is used.

10.2 Column Degradation Evaluation (PEM)

10.2.1 The column evaluation mix must be injected before each initial calibration, the beginning of an analytical sequence and every subsequent 12 hours of continuous analysis.

10.2.2 The degradation of DDT and Endrin must be calculated and each shown to $\leq$ 15% before calibration can proceed.

10.2.3 If the breakdown of DDT and/or Endrin exceeds the limits given above, corrective action must be taken. This action may include:

10.2.3.1 Replacement of the injection port liner or the glass wool.

10.2.3.2 Cutting off a portion of the injection end of a capillary column.

10.2.3.3 Replacing the GC column.

10.3 Initial Calibration

10.3.1 Prepare a Pesticide standard at a minimum of five concentration levels. (Six points are required if a quadratic (second order) curve is used.) The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Six points are required for quadratic (2nd order) curves.

10.3.2 A single point calibration for Technical Chlordane and Toxaphene is performed with every initial calibration for pattern recognition.

10.3.2.1 Select 3-5 major peaks in the multi-component analyte pattern. The area of each peak is used to calculate the concentration for each peak. An average of the concentrations is used.

10.3.2.2 If Toxaphene or Technical Chlordane is detected, sample will require re-analysis under a five point calibration for the analyte found. See Client Requirements Sheet to determine if this is needed.

10.3.3 The analyst may include a full 5 point calibration for any of the multi-component analytes with the initial calibration.

10.3.4 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., ECD replacement).

10.3.5 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the corporate policy, "Selection of Calibration Points", P-T-001.

10.3.6 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).

10.4 SW 8081 criteria

10.4.1 SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.

10.4.1.1 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.

10.4.1.2 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.

10.4.2 Linear calibration using the average response factor

10.4.2.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq$ 20% for each target analyte.

10.4.2.2 If the %RSDs in the initial calibration is > 20%, then calibration using a linear regression may be employed.

10.4.3 Linear calibration using a least squares regression

10.4.3.1 The intercept of the curve at zero response must be less than ± the reporting limit for the analyte.

10.4.3.2 r (correlation coefficient) must be $\geq$ 0.995 OR r^2 (coefficient of difference) must be $\geq$ 0.990.

10.4.3.3 Linear calibration using a least squares regression, forcing thru zero

10.4.3.3.1 Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression though zero for some calibrations.

10.4.3.3.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.

10.4.3.3.3 For samples requiring adherence to SW846 Method 8000B (i.e. South Carolina compliance work), forcing through zero is NOT allowed.

10.4.3.4 Linear calibration using a least squares regression, weighting of data points

10.4.3.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. For this reason it may preferable to increase the weighting of the lower concentration points. $^{1}/\text{Concentration}^{2}$ weighting (often called $^{1}/_{X}^{2}$ weighting) to improve accuracy at the low end of the curve.

10.4.3.4.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.

10.4.4 Non-linear calibration

10.4.4.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed. Non linear calibration requires 6 points.

10.4.4.1.1 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.4.4.2 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.

10.4.4.3 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990.

10.4.4.4 A quadratic calibration curve requires six standards.

10.4.4.5 Non-linear calibrations are NOT allowed for South Carolina compliance work.

10.5 608 Criteria

10.5.1 Method 608 only requires a 3-point calibration.

10.5.1.1 We routinely perform a 5-point calibration; however, 2 points may be removed from the curve if necessary to meet 608 calibration criteria.

10.5.1.2 Refer to the corporate policy, "Selection of Calibration Points", P-T-0001.

10.5.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 10\%$.

10.5.3 If the %RSDs in the initial calibration is > 10%, then calibration using a linear regression may be employed. See section 10.4.3 for criteria.

10.6 Initial Calibration Verification (ICV)

10.6.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.6.2 An ICV must be performed with every initial calibration.

10.6.3 The ICV performance must be within ± 20% D criteria.

10.6.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or

analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.7 Continuing Calibration Verification (CCV)

10.7.1 A CCV may be the same source or second source as the calibration.

10.7.2 Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a mid-level calibration standard.

10.7.3 SW 8081 criteria

10.7.3.1 The calibration verification is acceptable if the %D for each single component pesticide is ≤ 20%.

10.7.3.2 The same criteria will be used if Technical Chlordane or Toxaphene verifications are performed.

10.7.3.3 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).

10.7.4 608 criteria

10.7.4.1 At the beginning of each 24-hour long (or less) analytical sequence, demonstrate that the instrument calibration is still within the linear range established by the initial calibration through the analysis of mid-level standards of each compound of interest.

10.7.4.2 If the analyzed concentration of each analyte is within ± 15% of the true value, the daily calibration check is successful, and samples may be analyzed until 24-hours have elapsed from the injection of the first daily calibration standard.

10.8 Retention Time (RT) Windows

10.8.1 Retention Time (RT) windows must be determined for all analytes.

10.8.1.1 Establishing RT windows:

10.8.1.1.1 Make an injection of all analytes of interest each day over a three day period. Calculate the mean and the standard deviation of the three retention times for each analyte.

10.8.1.1.2 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater. Historically, calibrations RT windows have not been greater than 0.03 minutes. Windows larger than 0.03 minutes are indicative of equipment issues.

10.8.1.1.3 The center of the retention time window is the retention time from the CCV performed at the beginning of the analytical run. For samples run during the same shift as the initial calibration, use the retention time of the mid-point standard from the initial calibration. Some clients may have specific requirements regarding the updating of RT windows. Review the Client Requirement Memo for instructions.

10.8.1.1.4 Method 8000B requires a new retention time window study be performed annually or when the analytical column from a new vendor or different stationary phase is used. 8000C also requires a new study when the column is clipped during maintenance.

10.8.1.1.4.1 Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

10.8.2 Retention Time Criteria

10.8.2.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.9 Method Detection Limit Studies

10.9.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs.

11.0 PROCEDURE

11.1 Allow standards, samples and sample extracts to reach ambient temperature before analysis.

11.2 All analysis conditions and injection volumes for samples must be the same as for the calibration standards.

11.3 Sample Introduction

11.3.1 Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.4 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them at ≤6 °C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 External Standard Calculations

12.2.1 *Analyte Concentration (µg/L) in sample*

Concentration (µg/L):

$$[C] = \frac{A_x * V_t * D}{\overline{CF} * V_i * V_s}$$

Where:

$[C]$	=	*Analyte Concentration in sample (µg/L)*
A_x	=	*Area of peak (response)*
V_t	=	*Total volume of extract (µL)*
D	=	*Dilution factor*
$\overline{CF}$	=	*Calibration factor ($\overline{RF}$ in target) – Response factor*
V_i	=	*Volume of extract injected (µL)*
V_s	=	*Volume of sample extracted*

12.2.2 *On column concentration*

On Column Concentration (µg/mL):

$$[OC] = \frac{A_x}{\overline{CF}}$$

Where:

$[OC]$ = *On Column Concentration [typically expressed in µg/mL (ppm)]*

Then substitute/derive

$$[C] = [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

When *on column concentration* $[OC]$ is equal to the *CAL-AMT (calibration amount)* of the low level standard needed to support the *reporting limit (µg/L)* and we solve the equation for *concentration (µg/L)*

Then

$$[C] \equiv RL \equiv [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

Where:

RL = Reporting Limit

12.3 Breakdown calculations:

$$\textbf{\% breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD+DDE)}}{\text{sum of all peak areas (DDT+DDE+DDD)}} \times 100$$

$$\textbf{\% breakdown of endrin} = \frac{\text{sum of degradation peak areas (aldehyde+ketone)}}{\text{sum of all peak areas (endrin+aldehyde+ketone)}} \times 100$$

12.4 See TALS software for additional calculations.

12.5 Manual Integrations

12.5.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices. Manual integrations are denoted with a "M" flag on the Target quantitation report.

12.6 Dilutions

12.6.1 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.

12.6.2 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.

12.7 Carryover

12.7.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.7.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.7.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, carryover may have occurred.

12.7.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

12.7.1.3 If the chromatographic profile resembles the previous sample, the results are questionable.

12.7.1.3.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

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12.8.1 For method 8000C, report the lower result of the two columns, unless the Client SOW requires that the higher result be reported. See Client Requirements Sheet for determination. Method 8000B requires reporting the higher of the two columns.

12.8.1.1 For non-detect (ND) results, report from the A channel, unless there is evidence of chromatographic interference in the A channel's performance.

12.8.1.2 If one result is significantly higher (e.g., > 40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the lower result for 8000C and the higher result for 8000B. The data user should be advised of the disparity between the results on the two columns.

12.8.1.3 Use the higher result if there is obvious chromatographic interference on the column with the lower result.

12.8.1.3.1 If the CCV performance on one of the two channels is outside acceptance criteria due to confirmed matrix interference, report sample data from the column with acceptable performance, irrespective of it being the higher or lower result.

12.8.2 The QC should be reported from the column that reflects the column used for the majority of the samples associated with the QC.

12.8.3 The surrogate should be reported from the column that reflects the column used for the majority of the analytes associated with a sample.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet. For South Carolina compliance work, the Method

Blank must be below the RL. If not, perform necessary corrective action and re-analyse the associated samples.

13.2.1.3 The method blank must have acceptable surrogate recoveries.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – Blank contamination above the RL (>½ RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.2.2.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.3.1.1.1 For long analyte spike list, marginal exceedances (ME) are allowed as follows:

13.3.1.1.2 Marginal exceedance is not allowed for South Carolina compliance work, LCS recovery must fall within 70 -130% of the true value or corrective action taken and associated samples re-analyzed.)

13.3.1.1.3 < 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.

13.3.1.1.4 11 – 30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.

13.3.1.1.5 31 – 50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.

13.3.1.1.6 51 – 70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.

13.3.1.1.7 71 – 90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.

13.3.1.1.8 > 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.

13.3.1.1.9 No LCS recoveries may be outside the Marginal Exceedance limit.

13.3.1.1.10 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

13.3.1.1.11 The LCS should have acceptable surrogate recoveries.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

13.3.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.3.2.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.

13.3.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD)
13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evident of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

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13.5 Surrogate
13.5.1 All Surrogates should be within established control limits for accuracy (%Recovery).
13.5.2 Corrective Action for Surrogate not meeting acceptance criteria:
13.5.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample result evaluation
13.6.1 Dilutions
13.6.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
13.6.1.2 Dilution: Sample – An NCM is written to document the reason for the dilution.
13.6.1.3 .Dilution: Surrogates(s) and/or Spike(s) diluted out – Dilution: Surrogate(s) and/or spike(s) diluted out – An NCM is written to document the reason for the dilution
13.6.2 Carryover
13.6.2.1 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be demonstrated to not have occurred.
13.6.2.1.1 The carryover affected analyses are not reported, unless specifically requested by the client or there was insufficient extract/sample remaining to reanalyze.
13.6.3 Insufficient Sample
13.6.3.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. An NCM is written to document the insufficient volume..

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.
14.2 Demonstration of Capability
14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification
14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".

16.2.1.2 Vials containing sample extract will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "C".

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B and 8000C.

17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8081B.

17.3 USEPA Wastewater Method 608.

17.4 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.6 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices

17.7 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

17.8 Associated SOPs, current revisions;

17.8.1 ST-OP-0001, Labware Preparation for Organic Analysis

17.8.2 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series

17.8.3 ST-PM-0002, Sample Receipt and Chain of Custody

17.8.4 ST-QA-0002, Standard and Reagent Preparation

17.8.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

17.8.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.8.7 ST-QA-0016, IDL/MDL Determination

17.8.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

18.1.1 The Method Blank MUST be below the RL for South Carolina compliance work.

18.2 The surrogate calibration curve is calculated from last curved that is run before samples are injected.

18.3 SW.8846 requires that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the sample the laboratory receives, and the number of times the GC column may require clipping, TestAmerica St. Louis does not perform a RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.

18.4 Method 608 only requires a 3 point calibration. The laboratory routinely performs a 5 point calibration.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Updated section 4.2 by removing Gel Permeation Chromatography (Method 3640) and Sulfur cleanup (Method 3660).

19.2 Rev 11:

19.2.1 Annual Review, No Changes.

19.3 Rev 12:

19.3.1 Annual Review, No Changes.

19.4 Rev 13:

19.4.1 Added new equipment and supplies to section 6.0.

19.4.2 Updated information regarding reagent log in section 7.0.

19.4.3 Updated instructions for initial calibrations in section 10.3.

19.4.4 Updated non-linear calibration point requirements in section 10.4.

19.4.5 Updated requirements for performing RT window studies.

19.5 Rev 14:

19.5.1 Added new safety concerns in section 5.2

19.5.2 Updated location of where GC column type information can be found in section 6.0

19.5.3 Added software information to Section 6

19.5.4 Added method blank and LCS composition to Section 9..

19.5.5 Updated initial calibration peak calculations in section 10.3.

19.5.6 Updated quadratic calibration curve requirements in section 10.4.4.

19.5.7 Removed references to QuantIMS and Clouseau – replaced with LIMS.

19.5.8 Added reporting limit calculations to Section 12.

19.5.9 Added specific corrective actions to Section 13.

19.6 Rev 15 (06/02/14)

19.6.1 Added location of instrument run conditions in section 6.9

19.6.2 Updated processing software in section 6.2

19.6.3 Updated information on PEM standard in section 7.6.1

19.6.4 Corrected requirements for breakdown in section 10.2.2

19.6.5 Corrected storage requirements for standard in section 11.4

19.6.6 Updated software for calculations in section 12.3

19.6.7 Updated information for surrogate calibration in section 18.2

19.6.8 Updated Table 1

19.6.9 Updated Initial Calibration Sequence

19.7 Revision 16 (9/16/14)

19.7.1 Section 10.2 removed the incorrect reference to the location of the breakdown calculation

19.7.2 Section 10.4 updated to note that non-linear calibrations are not allowed for South Carolina compliance samples

19.7.3 Section 18 updated to list requirements for South Carolina regarding Method Blank acceptability

19.8 Revision 17 (10/31/14)

19.8.1 Calculations for breakdown products (DDT and endrin) added to Section 12.

19.8.2 Combined this SOP with requirements for South Carolina from SOP ST-GC-0016SC Rev 1 and retired that SOP.

19.9 Revision 18 (12/1/14) additional information for South Carolina added:

19.9.1 Columns used added to Section 6

19.9.2 MDL information added to Section 10CS specific limits added to Section 13

19.9.3 Standard and reagent information updated/added to Appendix 1. Appendix 2 added for preparation of working standards and calibration levels.

Table 1	
Column Degradation Evaluation Mix	
Component	Concentration (ug/L)
4,4'-DDT	100.000
Endrin	50.000

Analytical Sequence

Initial Calibration

Conditioning Standard	
Hexane Blank	
Instrument Blank	
Breakdown Mix (PEM)	
Individual mix	All levels
Individual mix ICV	
Technical Chlordane	All levels
Technical Chlordane ICV	
Toxaphene	All levels
Toxaphene ICV	
2,4'-DD*/HCB Mix (if needed)	All levels
2,4'-DD*/HCB Mix ICV (if needed)	
Sample injections (maximum 10)	
Continuing Calibration Verification (CCV)	Mid level (for needed target analytes)
Sample injections (maximum 10)	
Continuing Calibration Verification (CCV)	Mid level (for needed target analytes)

Breakdown check (beginning of an analytical run and subsequently every 12 hours of continuous analysis)

Appendix 1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8081tox_00016

Reagent ID: 8081tox_00016

Type:	ASTD	Expiration Date:	01/02/2015
Description:	Toxaphene	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101579
Creation Date:	06/11/2014	Vendor Cat #:	32071
Container(s):	395834		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Toxaphene					5000.00000	ug/mL
Toxaphene Peak 1					5000.00000	ug/mL
Toxaphene Peak 2					5000.00000	ug/mL
Toxaphene Peak 3					5000.00000	ug/mL
Toxaphene Peak 4					5000.00000	ug/mL
Toxaphene Peak 5					5000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8081mixab3SEC_00002

Reagent ID: 8081mixab3SEC_00002

Type:	ASTD	Expiration Date:	01/02/2015
Description:	indvidual mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A097850
Creation Date:	06/11/2014	Vendor Cat #:	32415
Container(s):	400232		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4,4'-DDD					2000.00000	ug/mL
4,4'-DDE					2000.00000	ug/mL
4,4'-DDT					2000.00000	ug/mL
Aldrin					2000.00000	ug/mL
alpha-BHC					2000.00000	ug/mL
alpha-Chlordane					2000.00000	ug/mL
beta-BHC					2000.00000	ug/mL
delta-BHC					2000.00000	ug/mL
Dieldrin					2000.00000	ug/mL
Endosulfan I					2000.00000	ug/mL
Endosulfan II					2000.00000	ug/mL
Endosulfan sulfate					2000.00000	ug/mL
Endrin					2000.00000	ug/mL
Endrin aldehyde					2000.00000	ug/mL
Endrin ketone					2000.00000	ug/mL
gamma-BHC (Lindane)					2000.00000	ug/mL
gamma-Chlordane					2000.00000	ug/mL
Heptachlor					2000.00000	ug/mL
Heptachlor epoxide					2000.00000	ug/mL
Methoxychlor					2000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8081mixab3_00002

Reagent ID: 8081mixab3_00002

Type:	ASTD	Expiration Date:	01/02/2015
Description:	indvidual mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101061
Creation Date:	06/11/2014	Vendor Cat #:	32415
Container(s):	400231		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4,4'-DDD					2000.00000	ug/mL
4,4'-DDE					2000.00000	ug/mL
4,4'-DDT					2000.00000	ug/mL
Aldrin					2000.00000	ug/mL
alpha-BHC					2000.00000	ug/mL
alpha-Chlordane					2000.00000	ug/mL
beta-BHC					2000.00000	ug/mL
delta-BHC					2000.00000	ug/mL
Dieldrin					2000.00000	ug/mL
Endosulfan I					2000.00000	ug/mL
Endosulfan II					2000.00000	ug/mL
Endosulfan sulfate					2000.00000	ug/mL
Endrin					2000.00000	ug/mL
Endrin aldehyde					2000.00000	ug/mL
Endrin ketone					2000.00000	ug/mL
gamma-BHC (Lindane)					2000.00000	ug/mL
gamma-Chlordane					2000.00000	ug/mL
Heptachlor					2000.00000	ug/mL
Heptachlor epoxide					2000.00000	ug/mL
Methoxychlor					2000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8081surr_00017

Reagent ID: 8081surr_00017

Type:	ASTD	Expiration Date:	11/30/2020
Description:	Pest Surr	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0105135
Creation Date:	10/30/2014	Vendor Cat #:	32000
Container(s):	490048		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
DCB Decachlorobiphenyl (Surr)					200.00000	ug/mL
Tetrachloro-m-xylene					200.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8081techchlor_00014

Reagent ID: 8081techchlor_00014

Type:	ASTD	Expiration Date:	03/31/2017
Description:	Technical Chlordane sec	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A094164
Creation Date:	04/30/2013	Vendor Cat #:	32072.sec
Container(s):	146584		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Technical Chlordane					5000.00000	ug/mL
Technical Chlordane Peak 1					5000.00000	ug/mL
Technical Chlordane Peak 2					5000.00000	ug/mL
Technical Chlordane Peak 3					5000.00000	ug/mL
Technical Chlordane Peak 4					5000.00000	ug/mL
Technical Chlordane Peak 5					5000.00000	ug/mL

APPENDIX 2

Standards:

Surrogate Stock – 200 ug/ml

Individuals Stock – 2000 ug/ml

Technical Chlordane Stock – 5000 ug/ml

Toxaphene Stock – 5000 ug/ml

Intermediates:

Surrogate Intermediate – 1 ml stock Q.S. to 10 ml with hexane = 20 ug/ml

Individuals Intermediate – 100 ul of stock Q.S. to 10 ml with hexane = 20 ug/ml

Technical Chlordane Intermediate - 1 ml of stock Q.S. to 5 ml with hexane = 1000 ug/ml

Toxaphene Intermediate – 1 ml of stock Q.S. to 5 ml with hexane = 1000 ug/ml

Working standards:

Individuals working – 250 ul of Individuals Intermediate + 250 ul of Surrogate Intermediate Q.S. with 50 ml hexane = 0.1 ug/ml Individuals / 0.1 ug/ml Surrogate

Technical Chlordane working – 50 ul of Technical Chlordane Intermediate Q.S. with 50 ml hexane = 1 ug/ml

Toxaphene working – 200 ul of Toxaphene Intermediate Q.S. with 50 ml hexane = 4 ug/ml

Calibration:
Toxaphene

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	20x	0.200
2	10x	0.400
3	4x	1.000
4	2x	2.000
5	1x	4.000
ICV (second source)	4x	1.000

Technical Chlordane

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	20x	0.050
2	10x	0.100
3	4x	0.250
4	2x	0.500
5	1x	1.000
ICV (second source)	4x	0.250

Indviduals and surrogate

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	20x	0.005
2	10x	0.010
3	4x	0.025
4	2x	0.050
5	1x	0.100
ICV (second source)	4x	0.025

Title: ANALYSIS OF DISSOLVED GASES IN GROUNDWATER

[Modified Method EPA RSK-175 (GC-FID)]

Approvals (Signature/Date):

Dennis Konopka GC Department Lead 5/30/14 Date	Michael Ridenhower Health & Safety Manager / Coordinator 5/30/14 Date
Tony L. Byrd Quality Assurance Specialist 5-30-14 Date	Elaine Wild Laboratory Director 6/2/14 Date

This SOP was previously identified as SOP No. ST-GC-0019 Rev. 1

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1. SCOPE AND APPLICATION

1.1 This document describes a procedure for the determination of dissolved gases in groundwater. The method is applicable to the preparation of water samples for the analysis of the headspace to quantify part-per-billion levels of dissolved gases in water samples.

1.2 This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

1.3 Analytes, Matrix(s), and Reporting Limits

1.3.1 The reporting limits and analytes are listed in Table 2.

1.3.2 If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

2. SUMMARY OF METHOD

2.1. A water sample is collected in the field in a 43-mL VOA vial with no headspace. Prior to analysis, the sample is transferred into a 22-mL serum vial with a crimp cap. Headspace is generated using UHP helium. The sample is loaded onto the headspace autosampler and analyzed by a Gas Chromatograph (GC) equipped with an FID Detector.

3. DEFINITIONS

3.1. See the TestAmerica St. Louis Quality Assurance Manual for a glossary of common laboratory terms and data reporting qualifiers.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them. There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, the analyst need to either turn off the power to the instrument, or disconnect it from its source of power.

5.3. There are no materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6. EQUIPMENT AND SUPPLIES

6.1 Sample Containers: 43-mL VOA vials 22 mL crimp cap vials

6.2 Instrumentation

6.2.1 Agilent 6890 GC with Flame Ionization Detector

6.2.2 Column (FID)- Restek RT-U-Plot and S-Plot; 30m, 0.53mm ID

6.2.3 Autosampler – Tekmar 7000 Headspace autosampler

6.3 Syringes: 10µL – 25.0-mL gas tight syringes

6.4 Data System – Chemstation for acquisition and Chrom for data processing.

6.5 Rotary or wrist Shaker or equivalent

6.6 Instrument run conditions can be found in the front of the maintenance calendars.

7. REAGENTS AND STANDARDS

7.1. Bulk tanks of ultrahigh purity nitrogen

7.2 Calibration Standards: The primary standard is purchased through Scott Gas. The calibration standard is comprised of one cylinders-- composed of nominally 1% (mole basis) Methane, Ethane, Ethene, Acetylene. The second source standard is a different lot provided by Scott Gas.

7.3 The calibration levels are achieved by using a syringe to inject different amounts of the standard into a 22-mL vial that contains 18 mL of deionized water and 4 mL of headspace.

7.4 Laboratory Control and Initial Calibration Verification Samples: The LCS and ICV are fortified to the concentration of a standard near the midpoint of the calibration curve.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for

detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples are collected in the field in a 43-mL screw cap VOA vial. The samples are preserved with 1:1 HCl to a pH of less than 2 where alkane analysis is required. Care should be taken that no headspace is present when capping the vials. A minimum of three vials per analysis are required. Double the volume for a sample with an MS/MSD. Samples are maintained at a temperature of 4 ± 2°C and should be analyzed within 14 days of collection.

8.3 If sample is unpreserved, analyze as is and document in the report. The holding time for unpreserved sample is 14 days.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD), which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents, the same processes, and the same personnel.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit, with the exception of common laboratory contaminants. Some clients and programs may require different method blank acceptance criteria. Review the Client Requirements Memo for specifics.

9.2.1.1. The common laboratory contaminant for this method is methane. This compound can be present up to five times the reporting limit.

9.2.2. Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, an NCM is generated and the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an impacted method blank, or the impacted anlayte(s) in the sample are 10X the concentration found in the blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS from an independent source must be processed with each preparation batch. The hydrocarbon LCS is fortified to the concentration of a standard near the midpoint of the calibration curve. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. Corrective Action for LCS

9.3.2.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.3.2.2. The only exception is if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. An NCM is written and the issue **must be addressed in the project narrative.**

9.3.2.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair is processed at client request for each batch of 20 samples. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. Corrective action for MS/MSDs

9.4.2.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.2.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is considered valueless.

9.4.2.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

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9.4.2.4. If client program requirements specify to confirm matrix interference's, repreparation and reanalysis of the MS/MSD may be necessary.

9.5. Control Limits

9.5.1. Control limits for the LCS and the MS/MSD are initially set at 40-150% with a 20% RPD. Limits will be based on statistical recovery once sufficient data points are collected. If insufficient sample is received for an MS/MSD and LCSD will be analyzed using the same RPD criteria as the MS/MSD of 20%.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified.

9.6. Nonconformance and Corrective Action

9.6.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration – Establish an initial calibration curve using the concentrations noted in the table below. Initial calibration standards are prepared in the same fashion as client samples. Thus a sample can be directly compared to the calibration curve to determine final concentration without the need to employ complex partial pressure calculations. See Appendix 1 for an example derivation of an initial calibration point.

Compound	Cal Level 1	Cal Level 2	Cal Level 3	Cal Level 4	Cal Level 5	Cal Level 6	Cal Level 7
Volume Injected (uL)	5	25	50	100	200	500	1000
Acetylene	2.96	14.8	29.6	59.3	118	296	592
Ethane	3.42	17.10	34.2	68.4	137	342	684
Ethene	3.19	16.0	31.9	63.8	128	319	638
Methane	1.82	9.13	18.3	36.5	73.0	183	365

** Concentrations are in µg/L unless noted otherwise.*

10.1.1. For each analyte, calculate the mean calibration factor from analyses of the calibration solutions.

10.1.2. Calculate the standard deviation (SD) and relative standard deviation (RSD) from each mean.

10.1.3. In general, for environmental analysis, average response factors are the most appropriate calibration model. If the average response factor is used if the average percent relative standard deviation (%RSD) for all analytes must be ≤ 20%. A linear curve can also be used with correlation coefficient r ≥ 0.990 for organics.

10.1.4. Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of a weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be less than the reporting limit.

Also examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered. A second order regression requires at least six calibration points. The linear regression must have a correlation coefficient (r) ≥ 0.990 for organics.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.1.5. Removal or replacement of levels from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below.

10.1.6. If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run, the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve re-analyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

10.1.7. One of the following conditions must be satisfied to allow removal or replacement of a level.

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For external standard methods, the average amount of analyte recovered is less than 70% or greater than 130% of the expected value.

10.1.8. The reason for replacing the level **must** be documented. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

10.1.9. Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed, then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event, the number of levels remaining in the calibration must be at least that required by the method.

10.1.10. Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method

10.2 Continuing Calibration Verification – Analyze a continuing calibration verification at the beginning of each 24-hr analytical window and every 10 samples. The CCV is fortified to the concentration of a standard near the midpoint of the calibration curve. The analyzed values should be within 20% of the expected value. The data quality objective for the continuing calibration check standards is 80-120% The %D between the CCV CF and the calibration average CF for each analyte must be less than or equal to 20%.

10.3 Initial Calibration Verification - An ICV or second source must be analyzed following the acquisition of the initial calibration. The ICV is fortified to the concentration of a standard near the midpoint of the calibration curve. The calibration factor (CF) for the ICV must be within 20% D of that from the initial calibration.

10.4 Retention Time (RT) Windows

10.4.1 Retention Time (RT) windows must be determined for all analytes

10.4.1.1 To establishing RT windows make an injection of all analytes of interest each day over a three day period. Calculate the mean and the standard deviation of the three retention times for each analyte.

10.4.1.1.1 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater. Historically, calibrations RT windows have not been greater than 0.03 minutes. Windows larger than 0.03 minutes are indicative of equipment issues.

10.4.1.1.2 The center of the retention time window is the retention time from the CCV performed at the beginning of the analytical run. For samples run during the same shift as the initial calibration, use the retention time of the mid-point standard from the initial calibration. Some clients may have specific requirements regarding the updating of RT windows. Review the Client Requirement Memo for instructions.

10.4.1.1.3 A new retention time window study is performed annually or when the analytical column from a new vendor or different stationary phase is used.

10.4.1.1.3.1 Until these standards have been run on the new column, the retention time windows from the old column may be used,

updated with the retention times from the new initial calibration.

10.5 Retention Time Criteria

10.5.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by the QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Analysis

11.3.1. Sample Analysis (GC-FID) - Remove the samples from the refrigerator and allow to come to room temperature

11.3.2. 18 ml of sample is removed by gastight syringe and transferred into a 22-ml vial and immediately sealed with a crimp cap..

11.3.3. The sample is then shaken to allow the gases to equilibrate between the headspace and the liquid phase.

11.3.4. Load the sample onto the Agilent headspace autosampler. The autosampler allows the sample's water and headspace phases to equilibrate at 40°C. 1mL of the sample headspace is injected directly onto the GC column where the target compounds, if present, are detected by FID. Acquire the data and process on Chrom. The instrument operating conditions are outlined below.

<u>Recommended GC Conditions</u>

Gas Flows:	60 mL/min
Carrier (Nitrogen):	40 mL/min
Oven Program:	40 °C (hold for 2.25 minutes) to 120 °C (15 °C/min) hold for 2 minutes, for a total run time of 9.44 minutes.

<u>Recommended FID Conditions</u>

FID Temp:	250 °C
Hydrogen Flow:	40 mL/min
Air Flow:	250 mL/min
Nitrogen Makeup:	25 mL/min

Recommended Headspace Auto sampler Conditions

Zone Temp	40 °C	Vial Pressurization time	1.0 min
Sample Loop Temp	80 °C	Sample Loop fill time	1.0 min
Transfer Line Temp	80 °C	Sample Loop Equil. time	0.05 min
GC Cycle time	14.0 min	Sample Injection time	0.30 min
Vial equilibration time	8.0 min		

11.4. Analytical Documentation

11.4.1. Record all analytical information in the analytical logbook/logsheet in "real time", which may be in an electronic format, including the analytical data from standards, blanks, LCS's, MS/MSD's, and any corrective actions or modifications to the method.

11.4.2. All standards are logged into the Reagent Log within the LIMS. All standards are assigned a unique number for identification by the LIMS Reagent Log.

11.4.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.4.4. Sample results and associated QC are uploaded into LIMS after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Calibration Factor for GC-FID/PDD

$$\mathbf{CF_\chi = A_\chi / C_\chi}$$

where:

CF_χ	=	Calibration factor of compound χ
A_x	=	Peak height or area
C_x	=	Concentration of target analyte χ in sample (ug/L)

12.2 Percent Difference for Calibration Factors

% D= (CFave - CFc / CFave) x 100

where:

CFave	=	Average CF for an analyte from the initial calibration
CFc	=	CF for an analyte from current check standard

12.3 Relative Standard Deviation

%RSD= (SD/ CFave) x 100

where:

CFave	=	Average CF for an analyte from the initial calibration
SD	=	Standard Deviation of average CFs for a compound

12.4 Sample Concentration in water

$C\chi = (A\chi/CFave) \times DF$

where:

$C\chi$	=	Concentration of target analyte χ in sample (ug/L)
$A\chi$	=	Peak area of analyte χ
CFave	=	Average calibration factor for an analyte from the calibration
DF	=	Dilution factor

12.5 Additional equations and calculations are listed in the Laboratory's quality Manual and the following SOPs: Calibration curves (General), CA-Q-S-005 and Selection of Calibration Points, CA-T-P-002.

12.6 Example calculation is included in Appendix 1.

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1. The data assessment and corrective action process is detailed through the LIMS Non-Conformance (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2. Sample result evaluation

13.2.1. Dilutions

13.2.1.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.2.2. Carryover

13.2.2.1. When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.2.2.2. If a sample is analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.2.2.3. If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the

previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.3. Insufficient Sample

13.3.1. For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative. The insufficient sample description is included in the NCM within the type defining the excursion.

14. METHOD PERFORMANCE & DEMONSTRATION OF CAPABILITY

14.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

14.2. Method performance data, reporting limits, and QC acceptance limits, are available in the LIMS.

14.3. Initial and continuing demonstrations of capability requirements are established in The ST-QAM.

14.4. Training Qualifications

14.4.1. The Manager/Supervisor has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14.4.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis; see requirements in the ST-QAM.

15. VALIDATION

15.1. Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16. WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

16.2. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to ST-HS-0001, "Waste Minimization Plan".

16.3. There are no waste streams produced when this method is carried out.

17. REFERENCES

17.1. RSK SOP-175, Revision 2, May 2004

17.2. TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current version.

17.3. Corporate Quality Management Plan (CQMP), current version.

17.4. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001 and TestAmerica Pittsburgh Facility Addendum and Contingency Plan, current version.

17.5. Acceptable Manual Integration Practices, CA-Q-S-002, current version.

17.6. Calibration Curves (General), CA-Q-S-005, current version.

17.7. Associated SOPs, current revisions:

17.7.1. ST-OP-0001, Labware Preparation for Organic Analysis
17.7.2. ST-PM-0002, Sample Receipt and Chain of Custody
17.7.3. ST-QA-0002, Standard and Reagent Preparation
17.7.4. ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
17.7.5. ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.7.6. ST-QA-0016, IDL/MDL Determination
17.7.7. ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.7.8. ST-QA-0038, Procedure for Compositing and Sub-sampling.

18. CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 SW846 requires that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the sample the laboratory receives, and the number of times the GC column may require clipping, TestAmerica St. Louis does not perform a RT study after clipping a column.

19. REVISION HISTORY

19.1. Revision 0

19.2. Revision 1:

19.2.1. Added text to Section 10.1 detailing that samples and the calibration standards are treated in the same manner.

19.3 Revision 2:

19.3.1 Added the location for instrument run conditions to Section 6.

19.3.2 Updated RT Windows procedure in Section 10.4

19.3.3 Updated analystical procedure, Section 11.3.

19.3.4 Updated Section 15.

19.3.5 Added method deviation regarding clipped columns and RT Windows to Section 18.

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Table 1: Compound Constants and Reporting Limits

Compound	Molecular Weight (g)
Methane	16.04
Ethane	30.07
Ethene	28.05
Acetylene	26.04

Table 2: Reporting Limits (RL)

Compound	RL (µg/L)
Methane	5.0
Ethane	5.0
Ethene	5.0
Acetylene	5.0

Appendix 1 – Example Calculation of ICAL Concentrations

This example is for methane at the 7th calibration level, where 1,000 µL of the stock standard (10,000 ppmV) is used.

The calculation is based on the **Ideal Gas Law: PV = nRT**

Where:

P	=	pressure in atmospheres (760 mm Hg = 1 atmosphere)
V	=	volume of gas in liters
n	=	number of moles of gas
R	=	gas constant, i.e., 08206 atm L / mol K
T	=	temperature in degrees Kelvin (T = °C + 273.15)

Calculations for the standards are based on 22 °C at 754 mm Hg, therefore

P	=	754/760 = 0.99211 atm
V	=	1 L of stock standard gas
R	=	0.08206
T	=	22 + 273.15 = 295.15 °K

Solving for **n**,

$$n = \frac{PV}{RT} = \frac{0.99211 atm \times 1L}{0.08206 atm \bullet L/mole \bullet K \times 296.15K} = 0.04082 \text{ moles of methane in 1 L standard}$$

***NOTE**: Daily variations in laboratory temperature and pressure create less than 1% variation.*

Concentration of methane calculation in the Level 7 ICAL standard:

$$\frac{0.001\ L\ N_2}{0.018\ L\ H_2O} \times \frac{0.01\ L\ CH_4}{1.0\ L\ N_2} \times \frac{0.04082\ mole\ CH_4}{1.0\ L\ CH_4} \times \frac{16.04\ g\ CH_4}{1.0\ mole\ CH_4} = 0.00036375\ g\ CH_4/L\ H_2O =$$

$$363.8\ \mu g/L \text{ methane concentration in Level 7 ICAL standard}$$

Where:

0.001 L N_2	=	Volume of gas (in L) used to prepare the aqueous standard.
0.018 L H_2O	=	Volume of water in which the standards are prepared.
0.01 L CH_4	=	Volume of methane in 1.0 L of the gas standard, which is a 1% methane mixture in N_2
0.04082 mole CH_4	=	Number of moles of methane in 1.0 L of methane as determined above
16.04 g CH4	=	Molecular weight of methane

TestAmerica St. Louis RSK-175 Dilution Calculation Table

Dilution Required	Dilution Formula
2	9 mL sample : 9 mL Reagent water
5	3.6 mL sample : 14.4 mL Reagent water
10	1.8 mL sample : 16.2 mL Reagent water
18	1 mL sample : 17 mL Reagent water
36	0.5 mL sample : 17.5 mL Reagent water
72	0.25 mL sample : 17.75 mL Reagent water

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Title: GC/MS SEMIVOLATILES ANALYSIS [SW-846 8270D; EPA 625]

Approvals (Signature/Date):

11-19-14	11/19/14
Jeff Winkler — Date	Mike Ridenhower — Date
Extractable Dept. Supervisor	Health & Safety Manager / Coordinator
11-19-14	11/19/14
Marti Ward — Date	Elaine Wild — Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. ST-MS-0001 Rev. 18

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.2 This SOP is based on SW-846 Method 8000B, 8000C and 8270D and EPA method 625.

1.3 The following compounds are documented in the method as problematic:

1.3.1 Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.

1.3.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.3.3 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.3.4 Hexachlorophene may not be amenable to analysis by this method.

1.4 N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from Diphenylamine.

1.5 3-Methylphenol cannot be separated from 4-Methylphenol by the conditions specified in this method.

1.6 Phthalic acid decomposes in the gas chromatographic inlet and cannot be distinguished from Phthalic anhydride.

1.7 Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be reported as Azobenzene.

1.8 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

1.8.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel. Continuous liquid-liquid extraction may also be used. Solid samples are extracted with methylene chloride / acetone using sonication. Waste dilution is used for organic or unusual matrix samples. The sample extract is concentrated to a volume of 1 mL, 5 mL or 10 mL, and analyzed by GC/MS. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electro impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Instead of scanning everything in a retention time range, SIM looks for specific ions (qualitative and quantitative) that are placed in retention time groups. The ions used for qualitative and quantitative purposes are the same for scan and SIM analysis. SIM is not allowed for South Carolina work, as the laboratory does not hold certification there for the SIM method.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 SIM –Selected Ion Monitoring

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.4 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 **SPECIFIC SAFETY CONCERNS OR REQUIREMENTS**

5.2.1 Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile, Silver Shield, or similar gloves must be used.

5.2.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.2.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: HP 6890/5973 - An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source. Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 1 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.1.1 Column: Restek RXI-5Sil MS, 30 meters, 0.25mm ID, 0.25 µm df

6.2 Data System:

6.2.1 ChemStation software system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the length of the chromatographic program.

6.2.2 Target software system allows the searching of any GC/MS data file for ions of a specified mass and plots such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software allows integrating the abundances in any EICP for a specified time or scan-number limit. Also, for the non-target compounds with a mass spectrum that meets the required criteria, software must be available that allows for the comparison of sample spectra against the reference library spectra.

6.2.3 Data Library: NIST05

6.3 Carrier gas: Ultra high purity helium

6.4 Instrument columns and run conditions are posted in the instrument maintenance calendar.

6.5 Amber vials. Crimp top seals

6.6 Disposal pipettes

6.7 Micro syringes- 10µL, 250µL, 500µL, 1000µL. Hamilton 1700 series, Agilent Gold Standard

6.8 Volumetric flasks, Class A

6.9 Analytical Balance, capable of weighing ± 0.01 grams.

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 See recipes for standards and QC samples in the LIMS Reagent Log program. See Appendix 1 of this SOP

7.3 At a minimum, a five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Table 3 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.4 An Internal Standard (IS) solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.5 Internal Standards are added to all standards and extracts to result in 40 ng injected onto the column. SIM Analysis Internal Standards are added to all standards and extracts to result in 4 ng injected onto the column.

7.6 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared.

7.7 ICV standards, NIST traceable:

7.7.1 The Semivolatile ICV standard is a second source from the calibration standard, where a second viable source is available.

7.7.2 ICV standard is prepared and stored in the same way as calibration standards.

7.8 Standards are to be refrigerated at $\leq 6^{o}$C when not in use. Refrigeration at less than -10°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least 6 months after opening.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Water samples are collected in amber glass, unpreserved and stored at 4 ± 2°C.

8.3 Soil samples are refrigerated at 4 ± 2°C.

8.4 The extraction holding time for Semivolatiles analysis in waters is 7 days.

8.5 The extraction holding time for Semivolatiles in soil/solid matrix is 14 days.

8.6 Extracts must be refrigerated at $\leq 6^{o}$C and analyzed within 40 days of the beginning of the extraction.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 DI water is used for the Method Blank.

9.2.4 Sodium sulfate is used as the method blank for solid matrices.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 The LCS is comprised of sodium sulfate fortified with the target analyte(s).

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 **Surrogate**

9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.

9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Internal standard calibration is used.

10.1.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 5. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.1.1.1 Compounds are assigned to the IS, generally with the closest retention time. See Table 5.

10.2 **Instrument Tuning**

10.2.1 The GC/MS system must be checked to see if acceptable performance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See Table 1 in this SOP.

10.2.1.1 The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

10.2.1.2 **8270** - At the beginning of every twelve hour shift.

10.2.1.3 **625** - At the beginning of every 24 hour shift.

10.2.1.3.1 The time period begins at the moment of injection of DFTPP.

10.2.2 Inject 50 ng of the GC/MS tuning standard into the GC/MS system. Obtain a background-corrected mass spectrum of DFTPP and confirm that all the key m/z criteria in Table 1 are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.2.3 Degradation of DDT to DDE and DDD should not exceed 20%.

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD \% DDE)}}{\text{sum of all peak areas (DDT \% DDE \% DDD)}} \times 100$$

10.2.4 Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

Tailing Factor = BC/AB

Where the peak is defined as follows:

AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height.

10.3 **Initial Calibration**

10.3.1 Prepare calibration standards at a minimum of five concentration levels, six points for a quadratic fit, (see Table 3 for suggested concentrations) for each parameter of interest. It may be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector.

10.3.2 Add the internal standard mixture to result in 40 ng on column. The concentrations of all analytes are listed in Table 3. Add the internal standard mixture to result in 4ng on column for SIM analysis.

10.3.3 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. The low level standard must be at or below the reporting limit.

10.3.4 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica corporate policy, "Calibration Curves."

10.3.5 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

10.3.6 A new calibration curve must be generated after major changes to the system and may be required when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., cleaning the ion source).

10.3.7 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).

10.3.8 **Initial Calibration Criteria (8270D)**

10.3.8.1 Minimum Response Factors

10.3.8.2 See Table 4 in this SOP for the minimum response factors. These minimum response factors are prescribed by SW method 8270D. For analytes not given a minimum response factor by the method, St. Louis has established a default minimum response factor of 0.01 for compound, except for Famphur, Hexachlorophene, Kepone and Phthalic Anhydride which have a minimum response factor of 0.001.

10.3.8.2.1 SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.

10.3.8.3 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.

10.3.8.4 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.

10.3.8.5 **Linear calibration using the average response factor**

10.3.8.5.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be ≤ 20% for each target analyte.

10.3.8.5.2 If the %RSD in the initial calibration is > 20%, then calibration using a linear regression may be employed.

10.3.8.6 **Linear calibration using a least squares regression**

. The intercept of a linear calibration at zero response (i.e. the y-intercept) must have an absolute value less than the reporting limit for each analyte. Client requirements may be tighter, please check Client Requirement Memorandum (CRM) if identified in comments.

Note, for analyses utilizing an internal standard the Target variable "b" does NOT equal the y-intercept. For analyses utilizing an internal standard, the Target variable "b" must be multiplied by the associated internal standard concentration to derive the concentration at the y-intercept.

10.3.8.6.1 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990.

10.3.8.6.2 When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve.

10.3.8.6.3 It is not necessary to re-analyze a low concentration standard; rather the data system can recalculate the concentrations.

10.3.8.6.4 The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

10.3.8.6.4.1 Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action should be taken.

10.3.8.7 **Linear calibration using a least squares regression, forcing thru zero**

10.3.8.7.1 Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression though zero for some calibrations.

10.3.8.7.2 Curve must still meet criteria in 10.3.8.6.1 and 10.3.8.6.2

10.3.8.7.3 For samples requiring adherence to method 8000B, forcing through zero is ***NOT*** allowed. This includes South Carolina compliance work.

10.3.8.8 **Linear calibration using a least squares regression, weighting of data points**

10.3.8.8.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration; for this reason it may be preferable to increase the weighting of the lower concentration points, $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting), to improve accuracy at the low end of the curve.

10.3.8.8.2 Curve must still meet criteria in 10.3.8.6.1 and 10.3.8.6.2

10.3.8.9 **Non-linear calibration**

10.3.8.9.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed.

10.3.8.9.2 The use of non-linear calibrations or second order regression calibrations are not allowed for South Carolina compliance samples.

10.3.8.9.3 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not to be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.3.8.9.3.1 These compounds are not to use non-linear calibrations: 1,4-Dioxane; Pyridine; n-Nitrosodimethylamine; 2-Fluorophenol; Aniline; Bis(2-chloroethyl)ether; Phenol-d5; Phenol; 2-Chlorophenol; 1,3-Dichlorobenzene; 1,4-Dichlorobenzene; 1,2-Dichlorobenzene; Benzyl Alcohol; 2-Methylphenol; N-nitrosodinpropylamine; Hexachloroethane; 3 and 4-Methylphenol; Nitrobenzene-d5; Nitrobenzene; Isophorone; 2-Nitrophenol; 2,4-Dimethylphenol; Bis(2-chloroethoxy) methane; 2,4- Dichlorophenol; 1,2,4-Trichlorobenzene; Naphthalene; Hexachlorobutadiene; 4-Chloro-3-Methylphenol; 2-Methylnaphthalene; 2,4,6-Trichlorophenol; 2-Fluorobiphenyl; 2,4,5-Trichlorophenol; 2-Chloronaphthalene; Dimethylphathalate; Acenaphthylene; Acenaphthene; Dibenzofuran; Diethylphthalate; Fluorene; 4-Chlorophenyl-phenylether; N-Nitrosodiphenylamine; Azobenzene; 4-Bromophenyl-phenylether; Hexachlorobenzene; Phenanthrene; Anthracene; Carbazole; Di-n-Butylphthalate; Fluoranthene; Pyrene; Terphenyl-d14; Butylbenzylphthalate; Benzo(a)Anthracene; Chrysene; bis(2-ethylhexyl)Phthalate; 2-Picoline; n-Nitrosomethylethylamine; Methyl methanesulfonate; n-Nitrosodiethylamine; Ethyl Methanesulfonate; Pentachloroethane; Acetophenone; n-Nitrosopyrrolidine; n-Nitrosomorpholine; O-Toluidine; n-Nitrosopiperidine; o,o,o-Triethyl-Phosphorothioate; 2,6-Dichlorophenol; Hexachloropropene; Benzothiazole; n-Nitrosodi-n-butylamine; Safrole; 1,2,4,5-Tetrachlorobenzene; cis-Isosafrole; trans-Isosafrole; 1,4-Dinitrobenzene; 1,3-Dinitrobenzene; Pentachlorobenzene; 1-Naphthylamine; 2-Naphthylamine; Thionazin; 5-Nitro-o-toluidine; Tri-n-butylphosphate; Sulfotepp; Diallate; Phorate; Phenacetin; Tris (2-chloroethyl) phosphate; 4-Aminobiphenyl; Pronamide; Pentachloronitrobenzene; Disulfoton; Parathion; Isodrin; Aramite; p- (Dimethylamino) azobenzene; Chlorobenzilate; 2-Acetylaminofluorene; 4,4'-Methylenebis (2)-Chloroaniline; 7,12-Dimethylbenz (a) anthracene; 3-Methylcholanthrene; Isosafrole; Octachlorostyrene; Methyl methacrylate; Ethyl methacrylate; Benzaldehyde; Caprolactam; 1-Methylnaphthalene; Biphenyl; Atrazine.

10.3.8.9.2.2 EPA Method 8000C suggests a 20% RSD limit be used when evaluating a calibration. The above compound list was constructed based on the 20% RSD criteria. TestAmerica St. Louis reserves the right to employ different calibration models when client mandated criteria are less than the 20% criteria found in method 8000C.

10.3.8.9.4 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.

10.3.8.9.5 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990.

10.3.8.9.6 Due to the nature of SIM analysis, non-linear calibrations may be used.

10.3.9 **625 criteria**

10.3.9.1 Method 625 only requires a 3 point calibration. We routinely perform a 6 point calibration; however, 3 points may be removed from the curve if necessary to meet 625 calibration criteria.

10.3.9.1.1 Refer to the TestAmerica corporate policy, "Calibration Curves."

10.3.9.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be < 35%.

10.3.9.3 If the %RSD in the initial calibration is > 35%, then calibration using a linear regression may be employed.

10.3.9.4 If a linear regression curve is used, the intercept of the curve at zero response must be less than ± the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.

10.3.9.5 Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

10.3.9.6 Weighting of data points

10.3.9.6.1 The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4 **<u>Initial Calibration Verification</u> (ICV)**

10.4.1 An initial calibration verification standard is a different standard source than the one used for the initial calibration.

10.4.2 An ICV must be performed with every initial calibration.

10.4.3 The ICV performance must be within ± 30% D criteria.

10.4.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.5 **<u>Continuing Calibration Verification</u> (CCV)**

10.5.1 At the start of each 12 hour period (8270) or 24 hour period (EPA 625) the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria. See Table 1 in this SOP.

10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.5.3 A CCV standard is analyzed every analysis tune clock immediately following the DFTPP tune.

10.5.3.1 **EPA 8270** – for each 12 hour tune time period

10.5.3.2 **EPA 625** – for each 24 hour tune time period

10.5.4 The CCV can be the same source or a second source from the calibration.

10.5.5 The internal standard response must be within 50-200 area counts (-50% to 100%) of the response in the mid level of the initial calibration. The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

10.5.6 **EPA 8270 Criteria**

10.5.6.1 Minimum Response Factors

10.5.6.2 See Table 4 in this SOP for the minimum response factors. These minimum response factors are prescribed by SW-846 method 8270D. For analytes not given a minimum response factor by the method, St. Louis has established a default minimum response factor of 0.01 per compound, except for Famphur, Hexachlorophene, Kepone and Phthalic Anhydride which have a minimum response factor of 0.001.

10.5.6.3 The CCV performance must be with ± 20% D criteria.

10.5.6.4 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).

10.5.7 **EPA 625 Criteria**

10.5.7.1 For each target analyte %D must be < 20%.

10.5.8 Calibration excursions are to be documented via a NCM.

10.6 Retention Time (RT) windows

10.6.1 Relative Retention Time (RRT)

10.6.1.1 In addition to normalizing the response (peak area) of the target compound to the response of the internal standard in that sample or extract for that injection, the retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound.

10.6.1.2 The RRT is expressed as a unit-less quantity:

$$RRT = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

10.6.1.3 The RRT of each target analyte in each calibration standard should agree within ± 0.06 RRT units.

10.6.1.4 It is recognized here that with increasing retention times of the internal standard, target analytes will be able to more easily meet this criterion. Thus, care should be exercised when selecting the appropriate internal standards by retention times. The process of selecting internal standards to quantify target analytes should also include consideration of retention times as they should be similar.

10.6.1.5 If this criterion is not met and unless there are no other indicators of a component's identification such as a very unique but a high probability mass spectral match then that component may not be considered as identified by relative retention time.

10.6.1.6 The RRT evaluation allows the analyst to compensate for modest shifts in the chromatographic conditions that can occur due to interferences and simple day-to-day instrument variability. Many methods that employ internal standard calibration use more than one internal standard and the target compounds are related to the internal standards on the basis of the similarity of their respective chromatographic retention times (see Table 5).

10.6.2 Internal standard retention time

10.6.2.1 The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.3 Retention Time Criteria

10.6.3.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.7 Method Detection Limit Studies

10.7.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

11.1 Samples are prepared following ST-OP-0002.

11.2 South Carolina requires a separate certification for SIM analysis. At this time TestAmerica St. Louis does not hold that certification. SIM analysis can not be used for South Carolina compliance samples.

11.3 South Carolina requires a separate certification for LVI (Limited Volume Extraction). At this time TestAmerica St. Louis does not hold that certification. LVI analysis can not be used for South Carolina compliance work.

11.4 All standards and extracts are allowed to warm to room temperature before injecting.

11.5 All samples must be analyzed using the same instrument conditions as the initial calibration.

11.6 Add internal standard to the extract to result in 40ng injected on column. Mix thoroughly before injection into the instrument.

11.6.1 Add internal standard to the extract to result in 4ng injected on column for SIM analysis.

11.7 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.8 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.9 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate at -10°C to -20°C (if it can be demonstrated that analytes do not fall out of solution at this temperature), protected from light in screw cap vials equipped with un-pierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 External Standard Calculations

12.1.1 See instrument software (Target/Chrom) for calculations.

12.2 Manual Integrations

12.2.1 Identified compounds are reviewed for proper integration. Integrations are performed automatically by the data system. If necessary, manual integrations are performed and are documented by the analyst. Manual integrations are denoted with “M” flag on the Target quantitation report. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices.

12.3 Qualitative identification

12.3.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify

identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

12.3.1.1 Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated. The following analytes should be carefully reviewed:

1,4-Dichlorobenzene-d4	Aniline	Bis (2-Chloroethyl) ether
1,3-Dichlorobenzene	1,4-Dichlorobenzene	1,2-Dichlorobenzene
Benzyl alcohol	2-Methylphenol	3,4-Methylphenol
2,4-Dichlorophenol	2,4,6-Trichlorophenol	2,4,5-Trichlorophenol
Phenanthrene	Anthracene	Benz (a) anthracene
Bis (2-ethylhexyl) phthalate	Chrysene	Di-n-octyl phthalate
Benzo (b) fluoranthene	Benzo (k) fluoranthene	Indeno (1,2,3-cd) pyrene
Benzo (g,h,i) perylene	p-Phenylenediamine	Safrole
Cis-Isosafrole	Trans-Isosafrole	1,4-Dinitrobenzene
1,3-Dinitrobenzene	1-Naphthylamine	2-Naphthylamine
2,3,4,6-Tetrachlorophenol	Dinoseb	Sulfotepp
Diallate 1 & 2	Methapyrilene	Aramite 1 & 2

12.3.2 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.3.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.3.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance should be between 20 and 80 percent.)

12.3.4.1 See Table 2 for primary, secondary and tertiary ion assignments.

12.3.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.3.6 Retention time criteria for samples

12.3.6.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

12.3.6.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

12.4 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client.

12.4.1 TICs are done as follows:

12.4.1.1 The computer will give quality matches in order from most likely to least likely. In order for us to call a TIC a certain compound, the quality match must be at least 90%. However, if the next two quality matches are within (around) 10% quality match of the first choice, the compound will be identified as an unknown because it is too close to call. Unknowns are put into a group if possible (such as Unknown alkanes) but if a group is not available it will be called Unknown. A compound will be also called unknown if the top three matches are all different groups of compounds and the quality match is < 90% (ex. If the top choice is an alkane, the second choice is an alcohol, the third choice is an acid).

12.4.1.2 The first 30 TICs, based on abundance, will be identified in a sample, unless a different number is specified by the client. See client requirement sheet.

12.5 Dilutions

12.3.7 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.

12.5.1 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.

12.5.2 Samples may be diluted initially if the project reporting limits are above the laboratory's routine calibration lower limit, if there is physical evidence of matrix, or historical knowledge of the site.

12.6 Carryover

12.6.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.6.1.1 If a sample analyzed after a sample with high concentrations has negative results or is non-detect, carryover did not occur.

12.6.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, carryover may have occurred.

12.6.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 This SOP lists requirements for the standard Quality Assurance criteria followed at TestAmerica St. Louis. If a client or program requires stricter quality controls (i.e. DoD, DOE, SC DHEC) the analyst is directed to the Client Requirement Memo for that client/project for limits.

13.2 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Steps taken for out-of-control situations include demonstrating that the cuase of the out-of-control situation was addressed and demonstration that a return to control was obtained.

13.3 Method Blank

13.3.1 Acceptance Criteria:

13.3.1.1 No target analytes may be present in the method blank above the reporting limit.

13.3.1.2 The method blank must have acceptable surrogate recoveries.

13.3.1.3 Corrective Action for Method Blanks not meeting acceptance criteria:

13.3.1.3.1 <u>Method Blank Contamination</u> – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.3.1.3.2 <u>Method Blank Surrogate excursion</u> – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-anlaysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.3.1.3.3 <u>For South Carolina compliance wrok, the Method Blank concentration must be below the RL</u>.

13.4 Laboratory Control Sample (LCS)

13.4.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).

13.4.1.1 For long analyte spike lists, marginal exceedances (ME) are allowed as follows:

13.4.1.2 less than 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.

13.4.1.3 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.

13.4.1.4 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.

13.4.1.5 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.

13.4.1.6 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.
13.4.1.7 More than 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.
13.4.1.8 No LCS recoveries may be outside the Marginal Exceedance limit.
13.4.1.9 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.
13.4.1.10 Marginal exceedance is not allowed by all programs. See Project/Program CRM for details. The use of marginal exceedances is not allowed for South Carolina Compliance samples.

13.4.2 The LCS should have acceptable surrogate recoveries.

13.4.3 Corrective Action for LCS not meeting acceptance criteria:

13.4.3.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. . In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.
13.4.3.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.
13.4.3.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.
13.4.3.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM.

13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.5.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

13.6 Sample result evaluation

13.6.1 Dilutions

13.6.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
13.6.1.2 Dilution: Sample– An NCM is created when dilutions are required.
13.6.1.3 Dilution: Surrogate(s)/spikes diluted out– An NCM is generated to document the surrogates/spikes being diluted out.

13.6.2 Carryover

13.6.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
13.6.2.2 If a sample analyzed after a sample with high concentrations is non-detect for the high concentration analyte, carryover did not occur.
13.6.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.6.3 Internal Standards

13.6.3.1 Acceptance Criteria:

13.6.3.1.1 If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in

the mid-point standard level of the most recent initial calibration sequence, corrective action must be taken.

13.6.3.1.2 If the EICP area for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified. (Some programs may require that the midpoint of the initial calibration be used for ISTD monitoring. See the project CRM for specifics.)

13.6.3.2 Corrective Action for Internal Standards not meeting acceptance criteria:

13.6.3.2.1 Internal Standard excursion – high – High ISTD recovery indicates a potential low bias to analytical results. Instrument maintenance, if required, is done and affected samples are reanalyzed. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.

13.6.3.2.2 Internal Standard excursion – low – Low ISTD recovery indicates the potential for a high bias to analytical results. Samples that are non-detect for affected analytes may be reported with an NCM. Samples with positive hits above the RL for analytes associated with the poor ISTD recovery require re-analysis. Instrument maintenance, if required, is done. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.

13.6.4 Surrogate

13.6.4.1 All Surrogates should be within established control limits for accuracy (%Recovery).

13.6.4.2 Corrective Action for Surrogate not meeting acceptance criteria:

13.6.4.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.7 Insufficient Sample

13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, an NCM is created and a narrative comment stating such is included in the report's Case Narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based

measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Auto-sample vials containing Methylene Chloride are to be disposed of in the appropriate solvent vial waste accumulation container located within the GC/MS lab, for temporary storage. Once this temporary container is full or once it reaches a one-year collection time, this container must be dumped into the permanent solvent vial waste container located in the 90-day storage area, which is marked as a Type "C" waste accumulation container.

16.2.1.2 Waste Methylene Chloride rinses are to be collected and disposed of within the solvent waste accumulation container located in the Organic Prep. Lab. This temporary storage container shall be dumped on a daily basis into the permanent waste accumulation container located in the 90-day storage area which is marked as a Type "D" waste drum.

17.0 REFERENCES

17.1 SW846, Test Methods for Evaluating Solid Waste, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Method 8000B, 8000C and 8270D.

17.2 40CFR Part 136: "Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", Code of Federal Regulations, Revised July1, 1995, Method 625.

17.3 TestAmerica St. Louis Quality Assurance Manual (QAM), current revision.

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-002) and St. Louis Facility Addendum (ST-HS-0002), current revision.

17.5 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices

17.6 TestAmerica Policy CA-T-P-002, Selection of Calibration Points

17.7 Associated SOPs, current revisions

17.7.1 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series Methods

17.7.2 ST-PM-0002, Sample Receipt and Chain of Custody

17.7.3 ST-QA-0002, Standard and Reagent Preparation

17.7.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.

17.7.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.7.6 ST-QA-0016, IDL/MDL, LOD/LOQ Determination

17.7.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

19.0 CHANGES TO PREVISION SOP REVISION

19.1 Table reference in Section 6.1 was corrected.
19.2 Y-intercept requirements added to Section 10.
19.3 Added requirement for 6 levels for a quadratic curve to Section 10
19.4 Added CLP allowance for reporting data within 10% of upper standard without dilution to Section 12
19.5 Clarification of criteria for TIC reporting added to Section 12.4.
19.6 Table 1: clarified Tune criteria and added allowance of other published DFTPP Tune criteria (i.e. EPA CLP)
19.7 Added Table 5, a listing of internal standards and associated analytes
19.8 Revision 13:
- 19.8.1 Grammatical /spelling corrections
- 19.8.2 Added SIM analysis to section 11

19.9 Revision 14:
- 19.9.1 Removed QuantIMS and Clouseau references – replaced with LIMs
- 19.9.2 Created hyperlinks to tables
- 19.9.3 Appended LVI Calibration Levels to Table 3
- 19.9.4 Combined fragmented Table 5 into one table
- 19.9.5 Added table of potentially mis-identifiable analytes to Section 12.3.
- 19.9.6 Removed CLP allowance for reporting data within 10% of upper standard without dilution from Section 12.
- 19.9.7 Revised Section 13 to remove Clouseau corrective action references and to provide specific corrective actions for non-conformances.

19.10 Revision 15:
- 19.10.1 Section 3, updated SIM definition
- 19.10.2 Section 7.5 Added SIM requirement
- 19.10.3 Section 7.7 ICV standard 2nd source where available to acquire
- 19.10.4 Section 10.1 corrected table references
- 19.10.5 Section 10.2 Added % breakdown calculation and added Benzidine and pentachlorophenol requirements
- 19.10.6 Section 10.3.8.9.2.1 removed compounds that are not to use non-linear calibration model
- 19.10.7 Removed 12.6.1.3
- 19.10.8 Section 13.6.2.3 removed chromatographic profile reference

19.11 Revision 16:
- 19.11.1 Section 1.1 Added a missing period
- 19.11.2 Section 10.2.1.2 Added a missing period
- 19.11.3 Section 10.2.2 Added a missing period
- 19.11.4 Section 10.5.2 Added a missing period
- 19.11.5 Section 12.2.1 Added a missing period
- 19.11.6 Section 13.4.3.1 Added a missing period
- 19.11.7 Section 13.4.3.4 Added a missing period
- 19.11.8 Section 10.3.8.2 Took comma out after Kepone and added the word “and”
- 19.11.9 Section 10.5.6.2 Took comma out after Kepone
- 19.11.10 Section 17.5 Changed policy number from 001 to 002
- 19.11.11 Section 2.1 Added possible concentration of 5 mL
- 19.11.12 Added Surrogates to Section 13

19.11.13 Table 3 – Changed calibration concentration levels

19.12 Revision 17:

19.12.1 Added note to Section 11 that South Carolina requires certification for SIM analysis.

19.12.2 Updated Section 13.4 to disallow the use of marginal exceedance for South Carolina compliance work.

19.12.3 Updated Section 10.3.8 to disallow the use of non-linear or second order calibrations for South Carolina compliance work.

19.13 Revision 18 (11/3/14):

19.13.1 Combined SOP with SOP ST-MS-0001SC Rev 1 to include the South Carolina DHEC requirements.

19.13.2 Table 7 added to SOP - containing information on standards and reagents.

19.14 Revsion 19 (12/1/14)

19.14.1 Added liquid-liquid continuous extraction to Section 2.1.

19.14.2 Added column used to Section 6

19.14.3 Updated Section 10.3 to clarify South Carolina requirements regarding forcing through zero

19.14.4 Added Section 10.7 to list MDL information

19.14.5 Added Section 11.3 to disallow LVI procedure for South Carolina compliance work

19.14.6 Added standard preparation and concentration information to the SOP appendix

Table 1
DFTPP Key Ions and Ion Abundance Criteria*

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

* Tune criteria in use is a combination of 8270C and 8270D which is more stringent than either method. Alternatively, other documented tuning criteria (e.g. EPA CLP) may be used provided method performance is not aversely affected.

Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

Primary Standard			
Analyte	**Primary**	**Secondary**	**Tertiary**
1,4 Dioxane	88	58	43
n-Nitrosodimethylamine	74*	42	44
Pyridine	79	52	—
Dimethylformamide	44	73	42
Cyclohexanol	57	82	67
2-Fluorophenol (Surrogate Standard)	112	64	63**
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	65
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	111
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	111
2-Methylphenol	108*	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	121
3&4-Methylphenol	107	108	79
n-Nitroso-di-n-propylamine	70	42	101
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107*	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123

Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

Primary Standard			
Analyte	**Primary**	**Secondary**	**Tertiary**
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54**
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	—
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	—
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153*	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	—
4-Nitrophenol	109*	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	105	51

Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

Primary Standard			
Analyte	**Primary**	**Secondary**	**Tertiary**
n-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332**	141
Azobenzene	77	51**	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	203
Benzidine	184	92	185
Pyrene	202	200	203
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

* primary/secondary and/or tertiary ions are switched from order in Method
** not listed in the method

Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Methyl methacrylate	69	41	39
Ethyl methacrylate	69	41	39
2-Picoline	93	66	92
n-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
2-Fluorophenol (Surrogate Standard)	112	64	63**
n-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Benzaldehyde	77	106	51
Phenol-d5 (Surrogate Standard)	99	42	71
Pentachloroethane	117	119	167
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
Acetophenone	105	77	120
n-Nitrosopyrrolidine	100	41	42
n-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	—
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
n-Nitrosopiperidine	114	42	55
O,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	—
Naphthalene-d8 (Internal Standard)	136	68	54**
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
Benzothiazole	135	108	69
Caprolactam	55	113	42
p-Phenylenediamine	108	80	—
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
Phthalic anhydride	104	76	50
1-methylnaphthalene	142	141	115

Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole, cis	162	104	131
2-Fluorobiphenyl (Surrogate Standard)	172	171	—
Isosafrole, trans	162	104	131
Biphenyl	154	153	152
1,4-Dinitrobenzene	168	75	50
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Acenaphthene-d10 (Internal Standard)	164	162	160
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	—
2-Naphthylamine	143	115	—
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	107	96	143
1,3,5-Trinitrobenzene	213*	75	120
2,4,6-Tribromophenol (Surrogate Standard)	330	332**	141**
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate 1	86	234	43
Diallate 2	86	234	43
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	170
Pentachloronitrobenzene	237	142	214
Phenanthrene-d10 (Internal Standard)	188	94	80
Pronamide	173	175	145
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	75
Parathion	109	97	291
Isodrin	193	66	195

Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Kepone	272	274	237
Methapyrilene	97	58**	—
Octachlorostyrene	308	343	154
Terphenyl-d14 (Surrogate Standard)	244	122	212
Aramite 1	185	319	—
Aramite 2	185	319	—
p-(Dimethylamino)azobenzene	120*	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	—
2-Acetylaminofluorene	181	180	223
Famphur	218	125	93
Chrysene-d12 (Internal Standard)	240	120	236
Hexachlorophene	196	198	209
7,12-Dimethylbenz(a)anthracene	256	241	120
Perylene-d12 (Internal Standard)	264	260	265
3-Methylcholanthrene	268	252	126

***** primary/secondary and/or tertiary ions are switched from order in Method
** not listed in the method

Table 3
Calibration Levels, Primary Standard, µg/mL[3]

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
1,4 Dioxane	1	2	5	10	20	30	40	50	60
Pyridine	1	2	5	10	20	30	40	50	60
n-Nitrosodimethylamine	1	2	5	10	20	30	40	50	60
Dimethylformamide	1	2	5	10	20	30	40	50	60
Cyclohexanol	1	2	5	10	20	30	40	50	60
Aniline	1	2	5	10	20	30	40	50	60
Phenol	1	2	5	10	20	30	40	50	60
Bis(2-chloroethyl)ether	1	2	5	10	20	30	40	50	60
2-Chlorophenol	1	2	5	10	20	30	40	50	60
1,3-Dichlorobenzene	1	2	5	10	20	30	40	50	60
1,4-Dichlorobenzene	1	2	5	10	20	30	40	50	60
Benzyl alcohol	1	2	5	10	20	30	40	50	60
1,2-Dichlorobenzene	1	2	5	10	20	30	40	50	60
2-Methylphenol	1	2	5	10	20	30	40	50	60
2,2'-oxybis(1-chloropropane)[1]	1	2	5	10	20	30	40	50	60
3&4-Methylphenol	1	2	5	10	20	30	40	50	60
n-Nitroso-di-n-propylamine	1	2	5	10	20	30	40	50	60
Hexachloroethane	1	2	5	10	20	30	40	50	60
Nitrobenzene	1	2	5	10	20	30	40	50	60
Isophorone	1	2	5	10	20	30	40	50	60
2-Nitrophenol	1	2	5	10	20	30	40	50	60
2,4-Dimethylphenol	1	2	5	10	20	30	40	50	60
Benzoic acid	1	2	5	10	20	30	40	50	60
bis(2-Chloroethoxy)methane	1	2	5	10	20	30	40	50	60
2,4-Dichlorophenol	1	2	5	10	20	30	40	50	60
1,2,4-Trichlorobenzene	1	2	5	10	20	30	40	50	60
Naphthalene	1	2	5	10	20	30	40	50	60
4-Chloroaniline	1	2	5	10	20	30	40	50	60
Hexachlorobutadiene	1	2	5	10	20	30	40	50	60

Table 3
Calibration Levels, Primary Standard, µg/mL[3]

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
4-Chloro-3-methylphenol	1	2	5	10	20	30	40	50	60
2-Methylnaphthalene	1	2	5	10	20	30	40	50	60
Hexachlorocyclopentadiene	1	2	5	10	20	30	40	50	60
2,4,6-Trichlorophenol	1	2	5	10	20	30	40	50	60
2,4,5-Trichlorophenol	1	2	5	10	20	30	40	50	60
2-Chloronaphthalene	1	2	5	10	20	30	40	50	60
2-Nitroaniline	1	2	5	10	20	30	40	50	60
Dimethyl phthalate	1	2	5	10	20	30	40	50	60
Acenaphthylene	1	2	5	10	20	30	40	50	60
3-Nitroaniline	1	2	5	10	20	30	40	50	60
Acenaphthene	1	2	5	10	20	30	40	50	60
2,4-Dinitrophenol	2	4	10	20	40	60	80	100	120
4-Nitrophenol	2	4	10	20	40	60	80	100	120
Dibenzofuran	1	2	5	10	20	30	40	50	60
2,4-Dinitrotoluene	1	2	5	10	20	30	40	50	60
2,6-Dinitrotoluene	1	2	5	10	20	30	40	50	60
Diethylphthalate	1	2	5	10	20	30	40	50	60
4-Chlorophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Fluorene	1	2	5	10	20	30	40	50	60
4-Nitroaniline	1	2	5	10	20	30	40	50	60
4,6-Dinitro-2-methylphenol	2	4	10	20	40	60	80	100	120
N-Nitrosodiphenylamine	1	2	5	10	20	30	40	50	60
Azobenzene[2]	1	2	5	10	20	30	40	50	60
4-Bromophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Hexachlorobenzene	1	2	5	10	20	30	40	50	60
Pentachlorophenol	1	2	5	10	20	30	40	50	60
Phenanthrene	1	2	5	10	20	30	40	50	60
Anthracene	1	2	5	10	20	30	40	50	60
Carbazole	1	2	5	10	20	30	40	50	60
Di-n-butyl phthalate	1	2	5	10	20	30	40	50	60
Fluoranthene	1	2	5	10	20	30	40	50	60

Table 3
Calibration Levels, Primary Standard, µg/mL[3]

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Benzidine	1	2	5	10	20	30	40	50	60
Pyrene	1	2	5	10	20	30	40	50	60
Butyl benzyl phthalate	1	2	5	10	20	30	40	50	60
3,3'-Dichlorobenzidine	1	2	5	10	20	30	40	50	60
Benzo(a)anthracene	1	2	5	10	20	30	40	50	60
Bis(2-ethylhexyl)phthalate	1	2	5	10	20	30	40	50	60
Chrysene	1	2	5	10	20	30	40	50	60
Di-n-octylphthalate	1	2	5	10	20	30	40	50	60
Benzo(b)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(k)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(a)pyrene	1	2	5	10	20	30	40	50	60
Indeno(1,2,3-cd)pyrene	1	2	5	10	20	30	40	50	60
Dibenz(a,h)anthracene	1	2	5	10	20	30	40	50	60
Benzo(g,h,i)perylene	1	2	5	10	20	30	40	50	60

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.
[3] Lower concentration standards may be analyzed on a project specific basis.

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Methyl methacrylate	1	2	5	10	20	30	40	50	60
Ethyl methacrylate	1	2	5	10	20	30	40	50	60
2-Picoline	1	2	5	10	20	30	40	50	60
n-Nitrosomethylethylamine	1	2	5	10	20	30	40	50	60
Methyl methanesulfonate	1	2	5	10	20	30	40	50	60
n-Nitrosodiethylamine	1	2	5	10	20	30	40	50	60
Ethyl methanesulfonate	1	2	5	10	20	30	40	50	60
Benzaldehyde	1	2	5	10	20	30	40	50	60

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Pentachloroethane	1	2	5	10	20	30	40	50	60
Acetophenone	1	2	5	10	20	30	40	50	60
n-Nitrosopyrrolidine	1	2	5	10	20	30	40	50	60
n-Nitrosomorpholine	1	2	5	10	20	30	40	50	60
o-Toluidine	1	2	5	10	20	30	40	50	60
n-Nitrosopiperidine	1	2	5	10	20	30	40	50	60
O,o,o-Triethyl-Phosphorothioate	1	2	5	10	20	30	40	50	60
A,a-Dimethyl-phenethylamine	1	2	5	10	20	30	40	50	60
2,6-Dichlorophenol	1	2	5	10	20	30	40	50	60
Hexachloropropene	1	2	5	10	20	30	40	50	60
Benzothiazole	1	2	5	10	20	30	40	50	60
Caprolactam	1	2	5	10	20	30	40	50	60
p-Phenylenediamine	1	2	5	10	20	30	40	50	60
n-Nitrosodi-n-butylamine	1	2	5	10	20	30	40	50	60
Safrole	1	2	5	10	20	30	40	50	60
Phthalic anhydride	1	2	5	10	20	30	40	50	60
1-Methylnaphthalene	1	2	5	10	20	30	40	50	60
1,2,4,5-Tetrachlorobenzene	1	2	5	10	20	30	40	50	60
Isosafrole, cis	.5	1	2.5	5	10	15	20	25	30
Isosafrole, trans	.5	1	2.5	5	10	15	20	25	30
Biphenyl	1	2	5	10	20	30	40	50	60
1,4-Dinitrobenzene	1	2	5	10	20	30	40	50	60
1,4-Naphthoquinone	1	2	5	10	20	30	40	50	60
1,3-Dinitrobenzene	1	2	5	10	20	30	40	50	60
Pentachlorobenzene	1	2	5	10	20	30	40	50	60
1-Naphthylamine	1	2	5	10	20	30	40	50	60
2-Naphthylamine	1	2	5	10	20	30	40	50	60
2,3,4,6-Tetrachlorophenol	1	2	5	10	20	30	40	50	60
5-Nitro-o-toluidine	1	2	5	10	20	30	40	50	60
Thionazin	1	2	5	10	20	30	40	50	60
1,3,5-Trinitrobenzene	1	2	5	10	20	30	40	50	60
Sulfotepp	1	2	5	10	20	30	40	50	60
Phorate	1	2	5	10	20	30	40	50	60

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Phenacetin	1	2	5	10	20	30	40	50	60
Diallate 1	.5	1	2.5	5	10	15	20	25	30
Diallate 2	.5	1	2.5	5	10	15	20	25	30
Dimethoate	1	2	5	10	20	30	40	50	60
4-Aminobiphenyl	1	2	5	10	20	30	40	50	60
Pentachloronitrobenzene	1	2	5	10	20	30	40	50	60
Pronamide	1	2	5	10	20	30	40	50	60
Disulfoton	1	2	5	10	20	30	40	50	60
2-sec butyl-4,6-dinitrophenol (Dinoseb)	1	2	5	10	20	30	40	50	60
Methyl parathion	1	2	5	10	20	30	40	50	60
4-Nitroquinoline-1-oxide	1	2	5	10	20	30	40	50	60
Parathion	1	2	5	10	20	30	40	50	60
Isodrin	1	2	5	10	20	30	40	50	60
Kepone	1	2	5	10	20	30	40	50	60
Famphur	1	2	5	10	20	30	40	50	60
Methapyrilene	1	2	5	10	20	30	40	50	60
Octachlorostyrene	1	2	5	10	20	30	40	50	60
Aramite 1	.5	1	2.5	5	10	15	20	25	30
Aramite 2	.5	1	2.5	5	10	15	20	25	30
p-(Dimethylamino)azobenzene	1	2	5	10	20	30	40	50	60
p-Chlorobenzilate	1	2	5	10	20	30	40	50	60
3,3'-Dimethylbenzidine	1	2	5	10	20	30	40	50	60
Hexachlorophene	10	20	50	100	200	300	400	500	600
2-Acetylaminofluorene	1	2	5	10	20	30	40	50	60
Dibenz (a,j)acridine	1	2	5	10	20	30	40	50	60
7,12-Dimethylbenz(a)anthracene	1	2	5	10	20	30	40	50	60
3-Methylcholanthrene	1	2	5	10	20	30	40	50	60
2-Fluorophenol (Surrogate Standard	1	2	5	10	20	30	40	50	60
Phenol-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Nitrobenzene-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2-Fluorobiphenyl (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2,4,6-Tribromophenol (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Terphenyl-d14 (Surrogate Standard)	1	2	5	10	20	30	40	50	60

Calibration Levels SIM Standard, ug/mL

Naphthalene	0.2	0.5	1.0	2.0	5.0	10.0
Acenaphthylene	0.2	0.5	1.0	2.0	5.0	10.0
Acenaphthene	0.2	0.5	1.0	2.0	5.0	10.0
Fluorene	0.2	0.5	1.0	2.0	5.0	10.0
Phenanthrene	0.2	0.5	1.0	2.0	5.0	10.0
Pyrene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(a)anthracene	0.2	0.5	1.0	2.0	5.0	10.0
Chrysene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(b)fluoranthene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(k)fluoranthene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(a)pyrene	0.2	0.5	1.0	2.0	5.0	10.0
Indeno(1,2,3-cd)pyrene	0.2	0.5	1.0	2.0	5.0	10.0
Dibenz(a,h)anthracene	0.2	0.5	1.0	2.0	5.0	10.0
Anthracene	0.2	0.5	1.0	2.0	5.0	10.0
Fluoranthene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(g,h,i)perylene	0.2	0.5	1.0	2.0	5.0	10.0
2-Methylnaphthalene	0.2	0.5	1.0	2.0	5.0	10.0

Table 3

LVI Calibration Levels, Primary Standard, µg/mL[3]

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
1,4 Dioxane	1	2	5	10	20	30	40	50	60
Pyridine	1	2	5	10	20	30	40	50	60

Table 3

LVI Calibration Levels, Primary Standard, µg/mL[3]

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
n-Nitrosodimethylamine	1	2	5	10	20	30	40	50	60
Dimethylformamide	1	2	5	10	20	30	40	50	60
Cyclohexanol	1	2	5	10	20	30	40	50	60
Aniline	1	2	5	10	20	30	40	50	60
Phenol	1	2	5	10	20	30	40	50	60
Bis(2-chloroethyl)ether	1	2	5	10	20	30	40	50	60
2-Chlorophenol	1	2	5	10	20	30	40	50	60
1,3-Dichlorobenzene	1	2	5	10	20	30	40	50	60
1,4-Dichlorobenzene	1	2	5	10	20	30	40	50	60
Benzyl alcohol	1	2	5	10	20	30	40	50	60
1,2-Dichlorobenzene	1	2	5	10	20	30	40	50	60
2-Methylphenol	1	2	5	10	20	30	40	50	60
2,2'-oxybis(1-chloropropane)[1]	1	2	5	10	20	30	40	50	60
3&4-Methylphenol	1	2	5	10	20	30	40	50	60
n-Nitroso-di-n-propylamine	1	2	5	10	20	30	40	50	60
Hexachloroethane	1	2	5	10	20	30	40	50	60
Nitrobenzene	1	2	5	10	20	30	40	50	60
Isophorone	1	2	5	10	20	30	40	50	60
2-Nitrophenol	1	2	5	10	20	30	40	50	60
2,4-Dimethylphenol	1	2	5	10	20	30	40	50	60
Benzoic acid	1	2	5	10	20	30	40	50	60
bis(2-Chloroethoxy)methane	1	2	5	10	20	30	40	50	60
2,4-Dichlorophenol	1	2	5	10	20	30	40	50	60
1,2,4-Trichlorobenzene	1	2	5	10	20	30	40	50	60
Naphthalene	1	2	5	10	20	30	40	50	60
4-Chloroaniline	1	2	5	10	20	30	40	50	60
Hexachlorobutadiene	1	2	5	10	20	30	40	50	60
4-Chloro-3-methylphenol	1	2	5	10	20	30	40	50	60
2-Methylnaphthalene	1	2	5	10	20	30	40	50	60
Hexachlorocyclopentadiene	1	2	5	10	20	30	40	50	60
2,4,6-Trichlorophenol	1	2	5	10	20	30	40	50	60
2,4,5-Trichlorophenol	1	2	5	10	20	30	40	50	60

Table 3

LVI Calibration Levels, Primary Standard, µg/mL[3]

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
2-Chloronaphthalene	1	2	5	10	20	30	40	50	60
2-Nitroaniline	1	2	5	10	20	30	40	50	60
Dimethyl phthalate	1	2	5	10	20	30	40	50	60
Acenaphthylene	1	2	5	10	20	30	40	50	60
3-Nitroaniline	1	2	5	10	20	30	40	50	60
Acenaphthene	1	2	5	10	20	30	40	50	60
2,4-Dinitrophenol	2	4	10	20	40	60	80	100	120
4-Nitrophenol	2	4	10	20	40	60	80	100	120
Dibenzofuran	1	2	5	10	20	30	40	50	60
2,4-Dinitrotoluene	1	2	5	10	20	30	40	50	60
2,6-Dinitrotoluene	1	2	5	10	20	30	40	50	60
Diethylphthalate	1	2	5	10	20	30	40	50	60
4-Chlorophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Fluorene	1	2	5	10	20	30	40	50	60
4-Nitroaniline	1	2	5	10	20	30	40	50	60
4,6-Dinitro-2-methylphenol	2	4	10	20	40	60	80	100	120
N-Nitrosodiphenylamine	1	2	5	10	20	30	40	50	60
Azobenzene[2]	1	2	5	10	20	30	40	50	60
4-Bromophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Hexachlorobenzene	1	2	5	10	20	30	40	50	60
Pentachlorophenol	1	2	5	10	20	30	40	50	60
Phenanthrene	1	2	5	10	20	30	40	50	60
Anthracene	1	2	5	10	20	30	40	50	60
Carbazole	1	2	5	10	20	30	40	50	60
Di-n-butyl phthalate	1	2	5	10	20	30	40	50	60
Fluoranthene	1	2	5	10	20	30	40	50	60
Benzidine	1	2	5	10	20	30	40	50	60
Pyrene	1	2	5	10	20	30	40	50	60
Butyl benzyl phthalate	1	2	5	10	20	30	40	50	60
3,3'-Dichlorobenzidine	1	2	5	10	20	30	40	50	60
Benzo(a)anthracene	1	2	5	10	20	30	40	50	60
Bis(2-ethylhexyl)phthalate	1	2	5	10	20	30	40	50	60

Table 3

LVI Calibration Levels, Primary Standard, µg/mL[3]

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Chrysene	1	2	5	10	20	30	40	50	60
Di-n-octylphthalate	1	2	5	10	20	30	40	50	60
Benzo(b)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(k)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(a)pyrene	1	2	5	10	20	30	40	50	60
Indeno(1,2,3-cd)pyrene	1	2	5	10	20	30	40	50	60
Dibenz(a,h)anthracene	1	2	5	10	20	30	40	50	60
Benzo(g,h,i)perylene	1	2	5	10	20	30	40	50	60

[1]2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.
[3]Lower concentration standards may be analyzed on a project specific basis.

LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Methyl methacrylate	1	2	5	10	20	30	40	50	60
Ethyl methacrylate	1	2	5	10	20	30	40	50	60
2-Picoline	1	2	5	10	20	30	40	50	60
n-Nitrosomethylethylamine	1	2	5	10	20	30	40	50	60
Methyl methanesulfonate	1	2	5	10	20	30	40	50	60
n-Nitrosodiethylamine	1	2	5	10	20	30	40	50	60
Ethyl methanesulfonate	1	2	5	10	20	30	40	50	60
Benzaldehyde	1	2	5	10	20	30	40	50	60
Pentachloroethane	1	2	5	10	20	30	40	50	60
Acetophenone	1	2	5	10	20	30	40	50	60
n-Nitrosopyrrolidine	1	2	5	10	20	30	40	50	60
n-Nitrosomorpholine	1	2	5	10	20	30	40	50	60
o-Toluidine	1	2	5	10	20	30	40	50	60
n-Nitrosopiperidine	1	2	5	10	20	30	40	50	60
O,o,o-Triethyl-Phosphorothioate	1	2	5	10	20	30	40	50	60
A,a-Dimethyl-phenethylamine	1	2	5	10	20	30	40	50	60

LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
2,6-Dichlorophenol	1	2	5	10	20	30	40	50	60
Hexachloropropene	1	2	5	10	20	30	40	50	60
Benzothiazole	1	2	5	10	20	30	40	50	60
Caprolactam	1	2	5	10	20	30	40	50	60
p-Phenylenediamine	1	2	5	10	20	30	40	50	60
n-Nitrosodi-n-butylamine	1	2	5	10	20	30	40	50	60
Safrole	1	2	5	10	20	30	40	50	60
Phthalic anhydride	1	2	5	10	20	30	40	50	60
1-Methylnaphthalene	1	2	5	10	20	30	40	50	60
1,2,4,5-Tetrachlorobenzene	1	2	5	10	20	30	40	50	60
Isosafrole, cis	.5	1	2.5	5	10	15	20	25	30
Isosafrole, trans	.5	1	2.5	5	10	15	20	25	30
Biphenyl	1	2	5	10	20	30	40	50	60
1,4-Dinitrobenzene	1	2	5	10	20	30	40	50	60
1,4-Naphthoquinone	1	2	5	10	20	30	40	50	60
1,3-Dinitrobenzene	1	2	5	10	20	30	40	50	60
Pentachlorobenzene	1	2	5	10	20	30	40	50	60
1-Naphthylamine	1	2	5	10	20	30	40	50	60
2-Naphthylamine	1	2	5	10	20	30	40	50	60
2,3,4,6-Tetrachlorophenol	1	2	5	10	20	30	40	50	60
5-Nitro-o-toluidine	1	2	5	10	20	30	40	50	60
Thionazin	1	2	5	10	20	30	40	50	60
1,3,5-Trinitrobenzene	1	2	5	10	20	30	40	50	60
Sulfotepp	1	2	5	10	20	30	40	50	60
Phorate	1	2	5	10	20	30	40	50	60
Phenacetin	1	2	5	10	20	30	40	50	60
Diallate 1	.5	1	2.5	5	10	15	20	25	30
Diallate 2	.5	1	2.5	5	10	15	20	25	30
Dimethoate	1	2	5	10	20	30	40	50	60
4-Aminobiphenyl	1	2	5	10	20	30	40	50	60
Pentachloronitrobenzene	1	2	5	10	20	30	40	50	60
Pronamide	1	2	5	10	20	30	40	50	60
Disulfoton	1	2	5	10	20	30	40	50	60

LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
2-sec butyl-4,6-dinitrophenol (Dinoseb)	1	2	5	10	20	30	40	50	60
Methyl parathion	1	2	5	10	20	30	40	50	60
4-Nitroquinoline-1-oxide	1	2	5	10	20	30	40	50	60
Parathion	1	2	5	10	20	30	40	50	60
Isodrin	1	2	5	10	20	30	40	50	60
Kepone	1	2	5	10	20	30	40	50	60
Famphur	1	2	5	10	20	30	40	50	60
Methapyrilene	1	2	5	10	20	30	40	50	60
Octachlorostyrene	1	2	5	10	20	30	40	50	60
Aramite 1	.5	1	2.5	5	10	15	20	25	30
Aramite 2	.5	1	2.5	5	10	15	20	25	30
p-(Dimethylamino)azobenzene	1	2	5	10	20	30	40	50	60
p-Chlorobenzilate	1	2	5	10	20	30	40	50	60
3,3'-Dimethylbenzidine	1	2	5	10	20	30	40	50	60
2-Acetylaminofluorene	1	2	5	10	20	30	40	50	60
Dibenz (a,j)acridine	1	2	5	10	20	30	40	50	60
Hexachlorophene	10	20	50	100	200	300	400	500	600
7,12-Dimethylbenz(a)anthracene	1	2	5	10	20	30	40	50	60
3-Methylcholanthrene	1	2	5	10	20	30	40	50	60
2-Fluorophenol (Surrogate Standard	1	2	5	10	20	30	40	50	60
Phenol-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Nitrobenzene-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2-Fluorobiphenyl (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2,4,6-Tribromophenol (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Terphenyl-d14 (Surrogate Standard)	1	2	5	10	20	30	40	50	60

Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010

Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.600
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachlorethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Naphthalene	0.700
4-Chloroanline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthanlene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010

Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
2,6-Dinitrotulene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010

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Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

TestAmerica St. Louis has established a default minimum response factor of 0.01 for compound not identified in this table, except for Famphur, Hexachlorophene, Kepone, Phthalic Anhydride which have a minimum response factor of 0.001.

Table 5

Semi-Volatile Internal Standards with Corresponding Analytes*

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
1,4-Dioxane	Acetophenone	cis-Isosafrole	5-Nitro-o-toluidine	Benzidine	Benzo(b)fluoranthene
Methyl methacrylate	N-Nitrosopyrrolidine	1,2,4,5-Tetrachlorobenzene	4,6-Dinitro-2-methylphenol	Pyrene	Benzo(k)fluoranthene
Pyridine	N-Nitrosomorpholine	Hexachlorocyclopentadiene	N-Nitrosodiphenylamine	Terphenyl-d14	7,12-Dimethyl benz(a)anthracene
N-Nitrosodimethylamine	O-Toluidine	2,4,6-Trichlorophenol	Tri-n-butyl phosphate	Aramite 1	Hexachlorophene
N,N-Dimethylformamide	Nitrobenzene-d5	2,4,5-Trichlorophenol	Azobenzene	Kepone	Benzo(a)pyrene
Ethyl methacrylate	Nitrobenzene	2-Fluorobiphenyl	Sulfotep	Aramite 2	3-methylcholanthrene
2-Picoline	N-Nitrosopiperidine	trans-Isosafrole	Diallate 1	p-(dimethylamino) azobenzene	Indeno (1,2,3-cd) pyrene
N-Nitrosomethylethylamine	Isophorone	Biphenyl	1,3,5-Trinitrobenzene	Chlorobenzilate	Dibenz(a,h)anthracene
Methyl methanesulfonate	2-Nitrophenol	2-Chloronaphthalene	Phorate	3,3'-Dimethylbenzidine	Benzo(g,h,i)perylene
2-Fluorophenol	2,4-Dimethylphenol	2-Nitroaniline	4-Bromophenyl phenyl ether	Butyl benzyl phthalate	
Cyclohexanol	Bis (2-chloroethoxy) methane	1,4-Naphthoquinone	Phenacetin	2-Acetylaminofluorene	
N-Nitrosodiethylamine	o,o,o-Triethylphosphorothioate	1,4-Dinitrobenzene	Diallate 2	Famphur	
Ethyl methanesulfonate	Benzoic acid	Dimethylphthalate	Hexachlorobenzene	Benzo (a) anthracene	
Benzaldehyde	2,4-Dichlorophenol	1,3-Dinitrobenzene	Dimethoate	4,4'-methylenebis (2-Chloroaniline)	
Phenol-d5	a,a-Dimethylphenethylamine	Acenaphthylene	Atrazine	3,3'-Dichlorobenzidine	
Phenol	1,2,4-Trichlorobenzene	2,6-Dinitrotoluene	Tris(2-chloroethyl) phosphate	Chrysene	
Aniline	Naphthalene	3-Nitroaniline	4-Aminobiphenyl	Bis (2-ethylhexyl) phthalate	
Pentachloroethane	4-Chloroaniline	Acenaphthene	Pentachlorophenol	Di-n-octyl phthalate	
Bis (2-chloroethyl) ether	2,6-Dichlorophenol	2,4-Dinitrophenol	Pronamide		
2-Chlorophenol	Hexachloropropene	4-Nitrophenol	Pentachloronitrobenzene		
1,3-Dichlorobenzene	Hexachlorobutadiene	Dibenzofuran	Phenanthrene		

Table 5

Semi-Volatile Internal Standards with Corresponding Analytes*

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
1,4-Dichlorobenzene	Benzothiazole	Pentachlorobenzene	Disulfoton		
1,2-Dichlorobenzene	Caprolactam	2,4-Dinitrotoluene	Anthracene		
Benzyl alcohol	N-Nitroso-di-n-butylamine	1-Naphthylamine	Dinoseb		
2-Methylphenol	p-Phenylenediamine	2-Naphthylamine	Carbazole		
Bis (2-chloroisopropyl) ether	4-Chloro-3-methylphenol	2,3,4,6-Tetrachlorophenol	Methyl parathion		
3,4-Methylphenol	Safrole	Diethylphthalate	Di-n-butyl phthalate		
N-Nitroso-di-n-propylamine	2-Methylnaphthalene	Fluorene	Parathion		
Hexachloroethane		4-Chlorophenyl phenyl ether	4-Nitroquinoline-1-oxide		
		Thionazin	Methapyrilene		
		4-Nitroaniline	Isodrin		
		2,4,6-Tribromophenol	Fluoranthene		

* ISTD assignment is based on instrument operating conditions and column type and may vary slightly from this listing.

Table 6a Acid Surrogates with Corresponding Analytes

2-Fluorophenol	Phenol-d5	2,4,6-Tribromophenol
none	Phenol 2-Clorophenol 2-Methylphenol 3,4-Methylphenol 2-Nitrophenol 2,4-Dimethylphenol Benzoic acid 2,4-Dichlorophenol 2,6-Dichlorophenol 4-Chloro-3-methyl -phenol	2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 2,4-Dinitrophenol 4-Nitrophenol 2,3,4,6-Tetrachlorophenol 4,6-Dinitro-2-methyl phenol Pentachlorophenol

Table 6b Base/Neutral Surrogates with Corresponding Analytes

Nitrobenzene-d5	**2-Fluorobiphenyl**	**Terphenyl-d14**
1,4-Dioxane	Benzothiazole	Phenanthrene
Methyl methacrylate	Caprolactam	Disulfoton
Pyridine	N-Nitrosodi-n-butylamine	Anthracene
N-Nitrosodimethylamine	p-Phenylenediamine	Dinoseb
Dimethylformamide	Safrole	Carbazole
Ethyl methacrylate	2-Methylnaphthalene	Methyl parathion
2-Picoline	1-Methylnaphthalene	Di-n-butyl phthalate
N-Nitrosomethylethylamine	cis-Isosafrole	Parathion
Methyl methanesulfonate	1,2,4,5-Tetrachlorobenzene	4-Nitroquinoline-1-oxide
Cyclohexanol	Hexachlorocyclopentadiene	Methapyrilene
N-Nitrosodiethylamine	trans-Isosafrole	Isodrin
Ethyl methanesulfonate	Biphenyl	Fluoranthene
Benzaldehyde	2-Chloronaphthalene	Benzidine
Aniline	2-Nitroaniline	Pyrene
Pentachloroethane	1,4-Naphthoquinone	Aramite 1 & 2
Bis (2-Chloroethyl) ether	1,4-Dinitrobenzene	Kepone
1,2-Dichlorobenzene	Dimethyl phthalate	p-(dimethylamino) azobenzene
1,3-Dichlorobenzene	1,3-Dinitrobenzene	

Table 6b Base/Neutral Surrogates with Corresponding Analytes

Nitrobenzene-d5	**2-Fluorobiphenyl**	**Terphenyl-d14**
1,4-Dichlorobenzene	Acenapthylene	Chlorobenzilate
Benzyl alcohol	2,6-Dinitrotoluene	3,3-Dimethylbenzidine
2,2'-oxybis (1-Chloro propane)	3-Nitroaniline	Butyl benzyl phthalate
Acetophenone	Acenapthene	2-Acetylaminofluorene
N-Nitrosopyrrolidine	Dibenzofuran	Famphur
N-Nitrosodinpropylamine	Pentachlorobenzene	Benz (a) anthracene
N-Nitrosomorpholine	2,4-Dinitrotoluene	4,4'-Methylenebis (2-Chloro -aniline)
o-Toluidine	1-Naphthylamine	3,3'-Dichlorobenzidine
Hexachloroethane	2-Naphthylamine	Chrysene
	Diethyl phthalate	

Nitrobenzene
N-Nitrosopiperidine
Isophorone
Bis (2-Chloroethoxy) Methane
O,o,o-Triethylphosphoro Thioate
A,a-Dimethylphenethyl Amine
1,2,4-Trichlorobenzene
Naphthalene
4-Chloroaniline
Hexachloropropene
Hexachlorobutadiene

Fluorene
4-Chlorophenyl phenyl ether
Thionazin
5-Nitro-o-toluidine
4-Nitroaniline
N-Nitrosodiphenylamine
Tri-n-butyl phthalate
Azobenzene
Sulfotepp
Diallate 1 & 2
1,3,5-Trinitrobenzene
Phorate
4-Bromophenyl phenyl ether

Bis (2-ethylhexyl) phthalate
Di-n-octyl phthalate
Benzo (b) fluoranthene
Benzo (k) fluoranthene
7,12-Dimethylbenz (a) anthracene
Hexachlorophene
Benzo (a) pyrene
3-methylcholanthrene
Indeno (1,2,3-cd) pyrene
Dibenz (a,h) anthracene
Benzo (g,h,i) perylene

Phenacetin

Hexachlorobenzene

Dimethoate

Atrazine
Tris(2-chloroethyl)phosphate
4-Aminobiphenyl
Pronamide

Pentachloronitrobenzene

APPENDIX 1

Standard preparations:

SCAN Intermediates

Internal Standard at 2000 ppm (I.S.): 1 mL to 2.5 = 800 ppm

8270:

8270 Surrogate at 5000 ppm: 0.4 mL to 10 mL CH2CL2 = 200 ppm
Benzoic Acid at 2000 ppm: 1 mL to 10 mL CH2CL2 = 200 ppm
Cyclohexanol at 2000 ppm: 1 mL to 10 mL CH2CL2 = 200 ppm
List 1 STD 1 at 1000 ppm: 2 mL to 10 mL CH2CL2 = 200 ppm
List 1 STD 2 at 2000 ppm: 1 mL to 10 mL CH2CL2 = 200 ppm
List 1 STD 7 at 2000 ppm: 1 mL to 10 mL CH2CL2 = 200 ppm
N,N-Dimethylformamide at 5000ppm: 0.4 mL to 10 mL CH2CL2 = 200 ppm

` SIM Intermediate

Internal Standard at 2000 (I.S.): 0.1 mL to 2.5 mL = 80 ppm
CAL mix 5 at 2000 ppm: 1 mL to 10 mL CH2CL2 = 200 ppm
200 ppm PAH intermediate: 1 mL to 10 mL CH2CL2 = 20 ppm
8270 Surrogate at 5000 ppm: 0.04 mL to 10 mL CH2CL2 = 20 ppm

Working Standards Levels from 200 ppm Intermediates 8270:

FV = 1mL for all levels, 800 ppm I.S. addition to each level of 0.05 mL = 40 ppm

Level 1: 1 ppm = 0.005 mL of 200 ppm intermediate
Level 2: 2 ppm = 0.01 mL of 200 ppm intermediate
Level 3: 5 ppm = 0.025 mL of 200 ppm intermediate
Level 4: 10 ppm = 0.05 mL of 200 ppm intermediate
Level 5: 20 ppm = 0.1 mL of 200 ppm intermediate
Level 6 (CCV): 30 ppm = 0.15 mL of 200 ppm intermediate
Level 7: 40 ppm = 0.2 mL of 200 ppm intermediate
Level 8: 50 ppm = 0.25 mL of 200 ppm intermediate
Level 9: 60 ppm = 0.3 mL of 200 ppm intermediate
ICV: 30 ppm = 0.05 of 2nd source ICV 200 ppm intermediate

Working SIM Standards Levels from 20 ppm Intermediates:

FV = 1mL for all levels, 80 ppm I.S. addition to each level of 0.05 mL = 4 ppm

Level 1: 0.1 ppm = 0.005 mL of 20 ppm intermediate
Level 2: 0.2 ppm = 0.01 mL of 20 ppm intermediate
Level 3: 0.5 ppm = 0.025 mL of 20 ppm intermediate
Level 4 (CCV): 1 ppm = 0.05 mL of 20 ppm intermediate
Level 5: 2 ppm = 0.1 mL of 20 ppm intermediate
Level 6: 5 ppm = 0.25 mL of 20 ppm intermediate
Level 7: 10 ppm = 0.5 mL of 20 ppm intermediate
ICV: 1 ppm = 0.05 mL of 2nd source ICV 20 ppm intermediate

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

SV IS Mix_00103

Reagent ID: SV IS Mix_00103

Type:	ASTD	Expiration Date:	06/30/2020
Description:	SV Internal Standard Mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Stora(	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0104707
Creation Date:	08/18/2014	Vendor Cat #:	31206
Container(s):	429301		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,4-Dichlorobenzene-d4					2000.00000	ppm
Acenaphthene-d10					2000.00000	ppm
Chrysene-d12					2000.00000	ppm
Naphthalene-d8					2000.00000	ppm
Perylene-d12					2000.00000	ppm
Phenanthrene-d10					2000.00000	ppm

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

List 1 Std 1_00005

Reagent ID: List 1 Std 1_00005

Type:	ASTD	Expiration Date:	02/13/2015
Description:	8270 List 1 Std #1 Megamix parent std	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Stora(	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101615
Creation Date:	08/11/2014	Vendor Cat #:	567672
Container(s):	423987		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,1'-Biphenyl					1000.00000	ppm
1,2,4,5-Tetrachlorobenzene					1000.00000	ppm
1,2,4-Trichlorobenzene					1000.00000	ppm
1,2-Dichlorobenzene					1000.00000	ppm
1,3-Dichlorobenzene					1000.00000	ppm
1,3-Dinitrobenzene					1000.00000	ppm
1,4-Dichlorobenzene					1000.00000	ppm
1,4-Dioxane					1000.00000	ppm
1-Methylnaphthalene					1000.00000	ppm
2,2'-oxybis[1-chloropropane]					1000.00000	ppm
2,3,4,6-Tetrachlorophenol					1000.00000	ppm
2,4,5-Trichlorophenol					1000.00000	ppm
2,4,6-Trichlorophenol					1000.00000	ppm
2,4-Dichlorophenol					1000.00000	ppm
2,4-Dimethylphenol					1000.00000	ppm
2,4-Dinitrophenol					2000.00000	ppm
2,4-Dinitrotoluene					1000.00000	ppm
2,6-Dinitrotoluene					1000.00000	ppm
2-Chloronaphthalene					1000.00000	ppm
2-Chlorophenol					1000.00000	ppm
2-Methylnaphthalene					1000.00000	ppm
2-Methylphenol					1000.00000	ppm
2-Nitroaniline					1000.00000	ppm
2-Nitrophenol					1000.00000	ppm
3 & 4 Methylphenol					1000.00000	ppm
3-Nitroaniline					1000.00000	ppm
4,6-Dinitro-2-methylphenol					2000.00000	ppm

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

List 1 Std 1_00005

Reagent ID: List 1 Std 1_00005

Type:	ASTD	Expiration Date:	02/13/2015
Description:	8270 List 1 Std #1 Megamix parent std	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Stora(	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101615
Creation Date:	08/11/2014	Vendor Cat #:	567672
Container(s):	423987		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4-Bromophenyl phenyl ether					1000.00000	ppm
4-Chloro-3-methylphenol					1000.00000	ppm
4-Chloroaniline					1000.00000	ppm
4-Chlorophenyl phenyl ether					1000.00000	ppm
4-Nitroaniline					1000.00000	ppm
4-Nitrophenol					2000.00000	ppm
Acenaphthene					1000.00000	ppm
Acenaphthylene					1000.00000	ppm
Acetophenone					1000.00000	ppm
Aniline					1000.00000	ppm
Anthracene					1000.00000	ppm
Azobenzene					1000.00000	ppm
Benzo[a]anthracene					1000.00000	ppm
Benzo[a]pyrene					1000.00000	ppm
Benzo[b]fluoranthene					1000.00000	ppm
Benzo[g,h,i]perylene					1000.00000	ppm
Benzo[k]fluoranthene					1000.00000	ppm
Benzyl alcohol					1000.00000	ppm
Bis(2-chloroethoxy)methane					1000.00000	ppm
Bis(2-chloroethyl)ether					1000.00000	ppm
Bis(2-ethylhexyl) phthalate					1000.00000	ppm
Butyl benzyl phthalate					1000.00000	ppm
Carbazole					1000.00000	ppm
Chrysene					1000.00000	ppm
Dibenz(a,h)anthracene					1000.00000	ppm
Dibenzofuran					1000.00000	ppm
Diethyl phthalate					1000.00000	ppm

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

List 1 Std 1_00005

Reagent ID: List 1 Std 1_00005

Type:	ASTD	Expiration Date:	02/13/2015
Description:	8270 List 1 Std #1 Megamix parent std	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Stora(	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101615
Creation Date:	08/11/2014	Vendor Cat #:	567672
Container(s):	423987		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Dimethyl phthalate					1000.00000	ppm
Di-n-butyl phthalate					1000.00000	ppm
Di-n-octyl phthalate					1000.00000	ppm
Fluoranthene					1000.00000	ppm
Fluorene					1000.00000	ppm
Hexachlorobenzene					1000.00000	ppm
Hexachlorobutadiene					1000.00000	ppm
Hexachlorocyclopentadiene					1000.00000	ppm
Hexachloroethane					1000.00000	ppm
Hexadecane					1000.00000	ppm
Indeno[1,2,3-cd]pyrene					1000.00000	ppm
Isophorone					1000.00000	ppm
Naphthalene					1000.00000	ppm
n-Decane					1000.00000	ppm
Nitrobenzene					1000.00000	ppm
N-Nitrosodimethylamine					1000.00000	ppm
N-Nitrosodi-n-propylamine					1000.00000	ppm
n-Octadecane					1000.00000	ppm
Pentachlorophenol					2000.00000	ppm
Phenanthrene					1000.00000	ppm
Phenol					1000.00000	ppm
Pyrene					1000.00000	ppm
Pyridine					1000.00000	ppm

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8270 Surr Std_00019

Reagent ID: 8270 Surr Std_00019

Type:	ASTD	Expiration Date:	02/28/2018
Description:	8270 Surrogate parent standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Winkler, Jeff S
Storage Location:	Semi-volatiles Standards Stora(	Vendor:	Restek
Reagent Volume:	5.000 mL	Vendor Lot #:	A093638
Creation Date:	08/28/2013	Vendor Cat #:	567685
Container(s):	198745		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
2,4,6-Tribromophenol					5000.00000	ppm
2-Fluorobiphenyl (Surr)					5000.00000	ppm
2-Fluorophenol					5000.00000	ppm
Nitrobenzene-d5					5000.00000	ppm
Phenol-d5					5000.00000	ppm
Terphenyl-d14					5000.00000	ppm

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Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS [SW-846 8260; EPA 624; DW 524.2]

Approvals (Signature/Date):

Andrew Buettner 11-20-14 Andrew Buettner Date Volatiles Organics Manager	Michael Ridenhower 11/20/14 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 11-20-14 Marti Ward Date Quality Assurance Manager	Elaine Wild 11/20/14 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-MS-0002 Rev. 22

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices.

1.2 This SOP is applicable to SW-846 method 8000B, 8000C, 8260B, 8260C, Drinking water method 524.2, and EPA 624.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 1 to 200 µg/L for 5 mL waters, 0.5 to 40 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for high-level soils.

1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

1.5.1 Additional compounds may be amenable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2 In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and flushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components that are detected with a mass spectrometer.

2.3 Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.4 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electro impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Instead of scanning everything in a retention time range, SIM looks for specific ions (qualitative and quantitative) that are placed in retention time groups. The ions used for qualitative and quantitative purposes are the same for scan and SIM analysis. In additiona to using the Scan mode, the laboratory also runs a SIM method for 1,4 dioxane. Details are available in Appendix 1 of this SOP. The SIM method is currently not allowed for South Carolina compliance work, as certification is required and the lab does not yet hold that certification.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 SIM – selected ion monitoring

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water or purchased HPLC water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Trip Blanks, prepared from reagent water, can serve as a check on conditions during transportation from the field to the laboratory.

4.3 Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

4.6 Methylene Chloride, Acetone, and 2-Butanone are potential laboratory contaminants. Concentrations up to five times the level observed in the method blank, in associated laboratory samples, may be attributed to the presence of these compounds in the laboratory.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 **SPECIFIC SAFETY CONCERNS OR REQUIREMENTS**

5.2.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.2.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 **PRIMARY MATERIALS USED**

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 Micro syringes- 1.0 µL, 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, 1000 µL. Hamilton 1700 series

6.2 Volumetric flasks, Class A

6.3 Analytical Balance, capable of weighing ± 0.001 g

6.4 Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.

6.5 Vials: 20 and 40 mL with Teflon lined screw caps

6.6 Spatula: Disposable wooden tongue depressors

6.7 Disposable pipettes

6.8 pH paper: Wide range and 0.3 to 2.3

6.9 Glass beads: Store in a drying oven.

6.10 Gas Chromatograph/Mass Spectrometer System:

6.10.1 Gas Chromatograph: Hewlett Packard GC 5890 and Agilent 6890 system with temperature programming.

6.10.2 Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorb heater.

6.10.3 Gas Chromatographic Capillary Columns:

6.10.3.1 Mass Spectrometer: Hewlett Packard 5970, 5972 and 5973 mass spectrometers capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode.

6.10.3.2 Columns used: Restek RTX-VMS, 30 meters, 0.25mm inside diameter, 1.4 µm film thickness; Restek RTX-VMS, 40 meter, 0.18mm insdie diameter, 1 µm film thickness

6.10.4 GC/MS interface: In general split/splitless injector are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.10.5 Instrument setup and operating conditions are maintained in each instruments daily maintenance calendar.

6.10.6 Data System:

6.10.6.1 ChemStation software system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.10.6.2 Target software system allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software allows integrating the abundances in any EICP between for a specified time or scan-number limit. Also, for the non-target compounds, software a mass spectrum that meets the required criteria when 50ng of 4-Bromofluorobenzene (BFB) are must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library.

6.10.5.2.1 Data Library: NIST 98

6.11 Carrier gas: Ultra high purity helium

6.12 Make up gas: Ultra high purity helium

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Methanol: Purge and Trap Grade

7.3 Water: HPLC grade or equivalent.

7.4 See recipes for standards and QC samples in the Reagents section of TALS.[Also examples included in Appendix 2 and 3.

7.5 Calibration Standards and Surrogates

7.5.1 Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at 0 to -20 °C.

7.5.2 Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer.

7.5.2.1 See SOP: ST-QA-0002 for expiration date criteria.

7.6 Initial calibration verification (ICV) standards are similar to calibration standards, but are from a completely different source.

7.7 Internal standards: Internal standards are added to all samples, standards, and blank analyses.

7.8 Tuning standard: A 25 ng/µL 4-Bromofluorobenzene standard is made up that will deliver 50 ng (or 25 ng) on column upon injection.

7.9 Sodium bisulfate, crystal

7.10 Ottawa sand

7.11 Ascorbic acid, ACS Reagent Grade, granular

7.12 Sodium Thiosulfate, ACS Reagent Grade, granular

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should review the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Aromatic volatiles water samples are preserved with 1:1 HCl and stored at 4 ± 2 °C. Analysis hold time is 14 days from collection.

8.3 Aqueous samples are stored in glass containers with Teflon lined septa at 4 ± 2 ^{o}C, with minimum headspace.

8.4 Soil samples are refrigerated at 4 ± 2 °C. Analysis hold time is 14 days from collection.

8.4.1 Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4 ±2 ^{o}C. The extracts are stored with minimum headspace.

8.5 For 5035 analysis

8.5.1 Solid samples, for low level analysis, may be field preserved with sodium bisulfate solution, or collected unpreserved using the Encore™ (or equivalent) sampler and shipped to the laboratory within 48 hours of sampling.

8.5.1.1 Following shipment back to the lab the soil is preserved with sodium bisulfate or the sample is extruded into an empty, clean sealed vial and frozen within 48 hours of sampling.

8.5.1.2 It is recommended that two Encore (or equivalent) samplers be used for each field sample position, to allow for any reruns than may be necessary.

8.5.2 Solid samples, for medium level analysis, may be field extracted with methanol, or collected unpreserved using the Encore (or equivalent)™ sampler and shipped to the laboratory within 48 hours of sampling.

8.5.2.1 It is recommended that two Encore (or equivalent) samplers be used for each field sample position, to allow for any reruns than may be necessary.

8.5.2.2 Solid samples – field extracted with methanol

8.5.2.2.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.5.2.2.2. Seal the bottle and attach a label.

8.5.2.2.3. Weigh the bottle to the nearest 0.01 g and note the weight on the label.

8.5.2.2.4. Ship with appropriate sampling instructions.

8.5.2.2.4.1. At client request, the methanol addition and weighing may also be performed in the field.

8.5.2.2.4.2. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.5.2.3 Solid samples – field extracted with methanol

8.5.2.3.1. When the samples are returned to the lab, extrude the (nominal) 5 g (or 25 g) sample into a pre-weighed VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.6 An additional sample is collected for percent moisture determination.

8.7 EPA 524.2 Sample Dechlorination and Preservation

8.7.1 If specified by the client that actual drinking water samples containing residual chlorine are to be analyzed by the laboratory, the following procedure shall be used:

8.7.1.1 Finished drinking water samples suspected of containing residual chlorine shall have 25 mg of ascorbic acid added to each 40 mL VOA vial prior to filling. If the requested target analytes are not gases at room temperature or are not listed in Table 7 of EPA Method 524.2, sodium thiosulfate is recommended to reduce residual chlorine (3 mg sodium thiosulfate for each 40 mL VOA vial).

8.7.1.2 If the sample containers are prepared by the laboratory for filling by the sample collector, the ascorbic acid or sodium thiosulfate will be placed into <u>unpreserved</u> VOA vials (do not mix the ascorbic acid or sodium thiosulfate with the HCl used for preservation).

8.7.1.3 It will be the sample collector's responsibility to add additional ascorbic acid or sodium thiosulfate if a diethyl-p-phenylenediamine (DPD) test kit indicates residual chlorine in excess of 5 mg/L (25 mg ascorbic acid or 3 mg sodium thiosulfate per each 5 mg/L of residual chlorine).

8.7.1.4 After filling the VOA vial, the sample collector should adjust the sample to pH < 2 by carefully adding two drops of 1:1 HCl for each 40 mL of sample. Samples should be sealed and mixed for one minute. (Note: If the sample is to be analyzed for trihalomethanes (THMs) only, the HCl preservation step may be omitted <u>if</u> sodium thiosulfate was used for dechlorination.)

8.7.1.5 If a sample foams vigorously when HCl is added, the sample should be discarded. Fill fresh VOA vials containing ascorbic acid or sodium thiosulfate, but do not acidify. A note should be placed on the chain-of-custody that is submitted with the samples that they were "not acidified". These samples have a 24 hold time if target analytes other than THMs are to be analyzed.

8.7.1.6 Samples should be maintained at $4 \pm 2\ ^{o}C$ until analysis.

8.8 At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab.

8.8.1 This is an older procedure based on method 5030.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), Matrix Spike (MS) and Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. MS/MSD must be performed for each separate QC code.

9.1.5 Unless medium level (i.e. an extraction batch) the blank, LCS and MS/MSD MUST run in the same 12 hour clock. The definition of a non-extraction batch is a maximum of 20 samples run in a 12 hr clock.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 For Water analyses, the method blank is comprised of HPLC water.

9.2.3 For Soil analyses, the method blank is comprised of glass beads, or Ottawa sand (required by South Carolina)

9.2.4 **For water and low soil method 8260 and 524.2 analyses**

9.2.4.1 A method blank must be analyzed with every 12 hour analytical clock and/or with each batch of 20 samples, whichever occurs first.

9.2.5 **For water method 624 analyses**

9.2.5.1 A method blank must be analyzed with every 24 hour analytical clock and with each batch of 20 samples, whichever occurs first..

9.2.6 **For medium level soil method 8260 analyses**

9.2.6.1 A method blank must be prepared with every medium level soil batch (20 or fewer samples of the same matrix). The medium level method blank is tied to an extraction and does not require repeated analysis with each sample analysis batch.

9.2.7 Method blank population studies shall be performed periodically. The data from such studies shall be used to identify systemic contamination issues.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 For Water analyses, the LCS is comprised of HPLC water fortified with Volatiles.

9.3.2.1 Under current methodology, the CCV may also serve as the LCS; however, due to limitations in the LIMS, the CCV and LCS should **never** be generated from the same injection.

9.3.3 For Soil analyses, the LCS is comprised of glass beads or Ottawa sand fortified with Volatiles. South Carolina requires the use of Ottawa sand.

9.3.4 **For method 8260 and 524.2 analyses**

9.3.4.1 A LCS must be analyzed with every 12 hour analytical clock and with each batch of 20 samples, whichever occurs first..

9.3.5 **For method 624 analyses**

9.3.5.1 A LCS must be analyzed with every 24 hour analytical clock and with each batch of 20 samples.

9.3.5.2 A LCS must be prepared with every high level soil batch (20 or fewer samples of the same matrix).

9.3.6 **For medium level soil method 8260 analyses**

9.3.7 An LCS must be prepared with every medium level soil batch (20 or fewer samples of the same matrix).

9.3.7.1 The medium level LCS is tied to an extraction and does not require repeated analysis with each sample analysis batch.

9.4 **Matrix Spike/Matrix Spike Duplicate**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 **8260** – A MS/MSD must be analyzed with every 12 hour analytical clock and with each batch of 20 samples, whichever occurs first..

9.4.3 **524.2** – Method 524.2 does not require a MS/MSD be performed though it may be requested by the client. In the absence of client QC criteria for this MS/MSD, the laboratory will use 70 – 130% recovery limits, and RPD of 20%, as advisory criteria.

9.4.4 **624** - A MS/MSD must be analyzed with every 24 hour analytical clock and with each batch of 20 samples, whichever occurs first.

9.5 **Surrogate**

9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.

9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Internal standard calibration is used.

10.1.1 Internal standard calibration is used. The internal standards are listed in this SOP. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard.

10.2 **<u>Instrument Tuning</u>**

10.2.1 Each GC/MS system must be hardware-tuned to meet the abundance criteria, listed in this SOP, for a maximum of a 50 ng (8260 and 624) and 25 ng (524.2) injection or purging of BFB. These criteria must be met

10.2.1.1 **8260** – for each 12 hour time period

10.2.1.2 **524.2** – for each 12 hour time period

10.2.1.3 **624** – for each 24 hour time period

10.2.1.3.1. The time period begins at the moment of injection of BFB.

10.2.2 Inject the method appropriate amount of GC/MS tuning standard into the GC/MS system. Obtain a background-corrected mass spectra of BFB and confirm that all the key m/z criteria in Table 2 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3 **Initial Calibration**

10.3.1 Prepare a multi-point calibration curve by fortifying 5 mL, or 25 mL depending on the required analysis, of HPLC water with incrementing concentrations of Volatiles standard.

10.3.2 The initial calibration contains a minimum of 5 points, for each target analyte (6 points are required for a quadratic fit). The low level standard must be at or below the reporting limit. The other standards define the working range of the detector, with the highest level standard establishing the linear range of the instrument.

10.3.2.1 Note 624 requires a minimum 3 point calibration. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector, with the highest level standard establishing the linear range of the instrument.

10.3.3 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance.

10.3.4 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.3.5 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).

10.3.6 **Initial Calibration Criteria (SW 8000B/8260B)**:

10.3.6.1 The % RSD of the calibration check compounds (CCC) must be less than 30%. CCC are as follows:

- Vinyl Chloride
- 1,1-Dichloroethene
- Chloroform
- 1,2-Dichloropropane
- Toluene
- Ethylbenzene

10.3.6.2 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.3.6.3 The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor.

10.3.6.3.1. A **minimum response factor of 0.01** must be achieved for all other volatile target analytes.

Compound	Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

10.3.6.4 If all %RSDs in the initial calibration are ≤ 15%, then all analytes may use average response factor for calibration.

10.3.6.5 The analyst should evaluate analytes with %RSD > 15%.

10.3.6.5.1. Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. For criteria regarding the removing calibration points from the curve, refer to STL policy P-T-001, Selection of Calibration Points.

10.3.6.6 For SW846 Method 8000B

10.3.6.6.1. If the average of all %RSDs in the calibration is > 15%, the analyst should consider instrument maintenance to improve the linearity of response.

10.3.6.6.2. When a linear regression curve is used, the intercept of the curve at zero response must be less than ± the reporting limit for the analyte. Client requirements may be tighter.(see Section 10.3.7.6.1).

10.3.6.6.3. If a linear regression curve is used, r must be ≥ 0.995.

10.3.6.6.4. When a linear model is employed use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve and to better control the intercept at zero response.

10.3.6.6.5. The regression may be forced through zero when using a linear model. South Carolina compliance work does NOT allow forcing through zero. See specific client requirements to determine if this technique is allowed for specific sample groups.

10.3.7 **Initial Calibration criteria (8000C/8260C and 524.2)**:

10.3.7.1 Minimum Response Factors (SW 8260C only)

10.3.7.1.1. See Table 4 in this SOP for recommended minimum response factors prescribed by method 8260. For analytes not given a minimum response factor by the method, TestAmerica St. Louis has established a default of 0.01 and 0.001 depending on the nature of the compound.

10.3.7.2 SW-846 8260C and 524.2 chromatographic methods allow the use of both linear and non-linear models for the calibration data.

10.3.7.3 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.

10.3.7.4 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear

calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.

10.3.7.5 **Linear calibration using the average response factor**

10.3.7.5.1. The Relative Standard Deviation (RSD) of the calibration points from the curve used must be ≤ 20% for each target analyte.

10.3.7.5.2. If the %RSD in the initial calibration is > 20%, then calibration using a linear regression may be employed.

10.3.7.6 **Linear calibration using a least squares regression**

10.3.7.6.1. The intercept of a linear calibration at zero response (i.e. the y-intercept) must have an absolute value less than the reporting limit for each analyte. Client requirements may be tighter, please check Client Requirement Memorandum (CRM) if identified in comments. Note, for analyses utilizing an internal standard the Target variable "b" does NOT equal the y-intercept. For analyses utilizing an internal standard, the Target variable "b" must be multiplied by the associated internal standard concentration to derive the concentration at the y-intercept.

10.3.7.6.2. r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990.

10.3.7.6.3. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve.

10.3.7.6.4. It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations.

10.3.7.6.5. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

10.3.7.6.5.1. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action should be taken.

10.3.7.7 **Linear calibration using a least squares regression, forcing thru zero**

10.3.7.7.1. Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression though zero for some calibrations.

10.3.7.7.2. Curve must still meet criteria in 10.3.7.6.1 and 10.3.7.6.2

10.3.7.8 **Linear calibration using a least squares regression, weighting of data points**

10.3.7.8.1. In a linear model, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. For this reason it may preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) to improve accuracy at the low end of the curve.

10.3.7.8.2. Curve must still meet criteria in 10.3.7.6.1 and 10.3.7.6.2.

10.3.7.9 **Non-linear calibration**

10.3.7.9.1. In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance

criteria, a quadratic model may be employed. All quadratic curves require a minimum of 6 pts.

10.3.7.9.2. It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not to be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.3.7.9.3. The intercept of the curve at zero response must be less than ± the reporting limit for the analyte. (Some clients may have tighter criteria; check Client Requirement Memos)

10.3.7.9.4. r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990.

10.3.7.9.5. The regression may be forced through zero when using a non-linear model. See specific client requirements to determine if this technique is allowed for specific sample groups.

10.3.7.9.6. The use of quadratic curves is not allowed for South Carolina compliance samples.

10.3.8 **624 Criteria**

10.3.8.1 Method 624 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from the curve if necessary to meet 624 calibration criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.3.8.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be < 35%.

10.3.8.3 If the %RSD in the initial calibration is ≥ 35%, then calibration using a linear regression may be employed.

10.3.8.3.1. If a linear regression curve is used, the intercept of the curve at small zero response must be less than ± the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.

10.3.8.4 Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

10.3.8.4.1. Weighting of data points

10.3.8.4.2. The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4 **<u>Initial Calibration Verification</u> (ICV)**

10.4.1 The initial calibration verification standard is a different standard source than the one used for the initial calibration

10.4.2 An ICV must be performed with each initial calibration.

10.4.3 The ICV performance must be within ± 30% D criteria for each analyte.

10.4.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard.

10.4.4 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.4.4.1 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.4.4.2 Variance among vendor supplied standards for a few compounds is not atypical for long analyte lists. All ICV failures that cannot be associated to laboratory error will require the immediate analysis of a third standard in an attempt to characterize the bias. If a third standard is not in the laboratory it must be ordered immediately.

10.5 **Continuing Calibration Verification.(CCV)**

10.5.1 At the start of each 12 hour period (8260) or 24 hour period (624) the GC/MS tuning standard must be analyzed. A 50ng injection of BFB must result in a mass spectrum for BFB which meets the criteria. See Table 2 in this SOP.

10.5.2 Following a successful BFB analysis, the continuing calibration standard(s) are analyzed. The standards must contain all volatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration

10.5.3 A CCV standard is analyzed every analysis tune clock immediately following the BFB tune.

10.5.3.1 **8260 and 524.2** – for each 12-hour tune time period

10.5.3.2 **624** – for each 24-hour tune time period

10.5.4 The CCV can be the same source or a second source from the calibration.

10.5.5 The internal standard response must be within -50 – 100% of the response in the mid level of the initial calibration. The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

10.5.6 8000B/8260B criteria:

10.5.6.1 The SPCC compounds must have a minimum response factor (see Initial Calibration SPCC criteria)

10.5.6.2 The percent difference of the CCC compounds compared to the the initial calibration must be $\leq$ 20%.

10.5.6.3 In addition, if CCC compounds are not part of the target list, the percent difference or drift of all analytes must be $\leq$ 20%, with allowance being made for up to six target compounds to have percent drift greater than 20%. Due to poor responses, the following compounds are allowed to have a %D > 60%, but less than 100%: Cyclohexane, 2-Chloroethyl vinyl ether, 2-Nitropropane, 1,4-Dioxane, Tetrahydrofuran, n-butanol and Isobutanol.

10.5.6.3.1 South Carolina does not allow this exception for poor responders. If the CCCanalytes are not in the target compound list all analytes must be with 30% difference or drift for calibration verification.

10.5.6.4 In addition, if any target analyte's %D is > 20%, the entire target analyte list must be averaged. The average %D must be ≤ 20%. [NOTE: Averaging the entire target analyte list (or using the "Gand Mean") is not allowed for South Carolina compliance work.]

10.5.6.5 If none of the identified CCCs are in the special calibration, the project specific target analytes %D must be < 20% or a maximum %D as agreed with the client. If there is a special project/client %D criteria it is noted on the client requirement sheet; otherwise a maximum 20% D is applied.

10.5.6.6 There are instances where a small subset of the routinely calibrated analytes are needed for analysis (e.g. dilutions or project with abbreviated target analyte lists). In cases where the target analytes for analysis constitutes less than half of the total number of analytes in the calibration standard, apply the following:

10.5.6.6.1. The SPCC compounds must have a response factor (see initial calibration SPCC criteria) if the SPCC is a target of interest.

10.5.6.6.2. The percent difference of the CCC compounds from the initial calibration must be ≤ 20%, if the CCC is a target of interest. In addition, the percent difference or drift of all analytes of concern must be ≤ 60%, with allowance being made for, Cyclohexane, 2-Chloroethyl vinyl ether, 2-Nitropropane, 1,4-Dioxan, Tetrahydrofuran, n-butanol and Isobutanol. These compounds are allowed to have a %D > 60% but less than 100%.

10.5.6.6.2.1. South Carolina does not allow this exception for poor responders. If the CCC analytes are not in the target compound list all analytes must be with 30% difference or drift for calibration verification.

10.5.6.6.3. For target analyte lists with more than 20 compounds: if any target analyte's %D is > 15%, the entire target analyte list must be averaged. The average %D must be ≤ 15%. [NOTE: This averaging is not allowed for South Carolina compliance work.]

10.5.6.6.4. For target analyte lists with less than 20 compounds: each target analyte %D must be < 15% or a maximum %D as agreed with the client.

10.5.7 **8260C criteria:**

10.5.7.1 The CCV performance must be with ± 20% D criteria.

10.5.7.2 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration)

10.5.8 **624 criteria**

10.5.8.1 Continuing calibration %D criteria is given in Table 5 of the Method. The column "Range for Q" is used to determine if CCV target analytes meet acceptance.

10.5.8.2 All target analytes must be within the limits prescribed.

10.5.9 **524.2 criteria**

10.5.9.1 For each target analyte %D must be less than or equal to 30%.

10.5.10 Calibration excursions are to be documented via a NCM.

10.6 Retention Time (RT) windows

10.6.1 Relative Retention Time (RRT)

10.6.1.1 In addition to normalizing the response (peak area) of the target compound to the response of the internal standard in that sample or extract for that injection, the retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound.

10.6.1.2 The RRT is expressed as a unit-less quantity:

$$RRT = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

10.6.1.3 The RRT of each target analyte in each calibration standard should agree within ± 0.06 RRT units.

10.6.1.4 It is recognized here that with increasing retention times of the internal standard, target analytes will be able to more easily meet this criterion. Thus,

care should be exercised when selecting the appropriate internal standards by retention times. The process of selecting internal standards to quantify target analytes should also include consideration of retention times as they should be similar.

10.6.1.5 If this criterion is not met and unless there are no other indicators of a component's identification such as a very unique but a high probability mass spectral match then that component may not be considered as identified by relative retention time.

10.6.1.6 The RRT evaluation allows the analyst to compensate for modest shifts in the chromatographic conditions that can occur due to interferences and simple day-to-day instrument variability. Many methods that employ internal standard calibration use more than one internal standard, and the target compounds are related to the internal standards on the basis of the similarity of their respective chromatographic retention times.

10.6.2 Retention Time Windows

10.6.2.1 The maximum retention time window is ± 0.45 minutes from the established retention time of the target analyte in the initial calibration.

10.6.2.1.1. Establishing this maximum retention time window, ensures that the RRT criteria of RRT ± 0.06 is achieved.

10.6.3 Internal standard retention time

10.6.3.1 The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.4 Retention Time Criteria

10.6.4.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.7 Method Detection Limit Studies

10.7.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

11.1 Screening

11.1.1 Screening samples is a semi-quantitative determination and is not intended to be use as a reportable analytical result.

11.1.2 Screening may be performed on either a GC-FID or GC/MS instrument. The instrument should be capable of detecting the compounds of concern but does not adhere to any method calibration criteria or analysis tune clock times, nor are there any batch QC (eg method blanks, LCS) requirements for screening samples.

11.1.3 Water samples are screened using a minimum 0.1 mL of samples and diluting with target analyte free water to a final volume of 5 mL. Greater dilutions may be taken if the sample is suspected to have significantly high concentration of target analytes or interferents.

11.1.4 For soil samples, 1 g of sample is place in a 40 mL VOA vial with 5 mL of target analyte free water. Greater dilutions may be taken if the sample is suspected to have significantly high concentration of target analytes or interferents.

11.1.5 Sample screening may be performed utilizing a heated or non-heated purging vessel.

11.2 Allow standards, samples and sample extracts to reach ambient temperature before analysis.

11.3 All analysis conditions and injection volumes for samples must be the same for the calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.1 Water, soil and medium level extract analyses are routinely performed by heated purge.

11.3.1.1 If desired, water, TCLP and methanol extracts may be performed using a non-heated purge.

11.3.1.2 If non-heated purge is desired, the calibration and all QC samples must also be performed utilizing a non-heated purge.

11.4 **Water Sample Preparation (5030B/C method)**

11.4.1 Transfer 5 mL or 25 mL sample to a VOA vial.

11.4.2 Transfer the 5 mL or 25 mL sample into an empty labeled 40 mL VOA vial.

11.4.3 Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution)

11.4.3.1 For routine TCLP samples use 0.5 mL of TCLP sample leachate with 4.5 mL reagent water and spike with surrogate and internal standards.

11.4.3.1.1.1 Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits)

11.4.3.2 For low level TCLP samples, use 5 mL of TCLP sample leachate and spike with surrogate and internal standard.

11.4.4 Prepare a method blank with 5 mL or 25 mL of HPLC water. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution)

11.4.5 Prepare a LCS with 5 mL or 25 mL of HPLC water. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution). Add 10 µL of 25 µg/mL spiking solution

11.4.5.1 For samples designated for MS/MSD analysis, add 10 µL of 25 µg/mL of spiking solution.

11.4.6 Check and document the pH the remaining sample.

11.4.6.1 Do not check pH prior to taking aliquot for analysis.

11.5 **Low-Level Soil Preparation (5035A method)**

11.5.1 If samples arrive unpreserved, the laboratory must, within 48 hours of collection, preserve samples with sodium bisulfate or extrude sample into a clean, empty, sealed VOA vial and freeze.

11.5.1.1 Check the preparation method code to determine preservation.

11.5.2 <u>If samples arrive in the Encore (or equivalent) sampler and require Sodium bisulfate preservation</u>:

11.5.2.1 Pre-weigh a labeled 40 mL VOA vial.

11.5.2.1.1 Label with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction.

11.5.2.2 Extrude the soil sample from the Encore (or equivalent) sampler into the VOA vial.

11.5.2.3 Weigh the vial to the nearest 0.01 g.

11.5.2.4 Record weight in appropriate TALS prep-batch.

11.5.2.5 Add a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of HPLC water.

11.5.2.5.1 Soils containing carbonates may effervesce when adding the sodium bisulfate solution. If this is the case, retrieve a second Encore sample plug, add 5 mL of water instead, and freeze at < 10 ^{o}C until analysis.

11.5.2.6 Seal the vial.

11.5.2.7 Add 10 µL each of surrogate and internal standard through the septum to each sample and QC.

11.5.2.7.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.

11.5.2.7.2 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10µL of surrogate to the vial.

11.5.2.7.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL of surrogate and 10 µL of spiking solution.

11.5.3 <u>If samples arrive 40 mL VOA vial already preserved with Sodium bisulfate</u>:

11.5.3.1 Add 10 ul each of surrogate and internal standard through the septum to each sample and QC.

11.5.3.1.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.

11.5.3.1.2 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10 µL each of surrogate and internal standard to the vial.

11.5.3.1.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL each of surrogate and internal standard and 10 µL of spiking solution.

11.5.4 <u>If samples arrive in the Encore (or equivalent) sampler and require freezing as preservation:</u>

11.5.4.1 Pre-weigh a labeled 40 mL VOA vial.

11.5.4.1.1 Label with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction.

11.5.4.2 Extrude the soil sample from the Encore (or equivalent) sampler into the VOA vial.

11.5.4.3 Weigh the vial to the nearest 0.01 g

11.5.4.4 Record weight in appropriate TALS prep-batch.

11.5.4.5 Seal the vial.

11.5.4.6 Freeze sample until time of analysis.

11.5.4.7 Add 5 mL of HPLC water.

11.5.4.8 Seal the vial.

11.5.4.9 Add 10 µL each of surrogate and internal standard through the septum to each sample and QC.

11.5.4.9.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.

11.5.4.9.2 Prepare a Method Blank using 5.0 g of glass beads and 5mL of HPLC grade water. Add 10 µL each of surrogate and internal standard to the vial.

11.5.4.9.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL each of surrogate and internal standard and 10 µL of spiking solution.

11.5.5 <u>If samples arrive in 40 mL VOA vial and require freezing as preservation</u>:

11.5.5.1 Freeze sample until time of analysis.

11.5.5.2 Add 5 mL of HPLC water

11.5.5.3 Seal the vial.

11.5.5.4 Add 10 µL each of surrogate and internal standard through the septum to each sample and QC.

11.5.5.4.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.

11.5.5.4.2 Prepare a Method Blank using 5.0g of glass beads and 5mL of HPLC grade water. Add 10 µL each of surrogate and internal standard to the vial.

11.5.5.4.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL each of surrogate and internal standard and 10 µL of spiking solution.

11.6 **Low-Level Soil Preparation (superceded 5030 method)**

11.6.1 See SOP ST-QA-0038 for the procedure for sub sampling.

11.6.2 Weigh 5 ± 0.05 g of the sample into a pre-weighed 40 mL glass labeled vial.

11.6.2.1 If the sample is suspected or known to have high concentrations of analytes, reduce the sample aliquot to 1.0 g

11.6.3 Record the weight.

11.6.4 Add 5 mL HPLC water

11.6.5 Seal the vial.

11.6.6 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.6.7 Add 10 µL of surrogate standard through the septum.

11.6.7.1 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10 µL of surrogate to the vial.

11.6.7.2 Prepare a LCS using 5.0g glass beads, 10 µL of surrogate and 10 µL of spiking solution.

11.6.7.3 For samples designated as for MS/MSD, add 10 µL of spiking solution.

11.7 **Methanol Extraction of Soils (5035A method)**

11.7.1 Extrude the (nominal) 5 g sample into a pre-weighed VOA vial containing 5mL methanol (25mL methanol for the 25 g sampler).

11.7.2 Obtain the weight of the soil added to the vial and note in preplog (Attachment 1).

11.7.3 Add 5 µL medium level surrogate to each sample and QC samples.

11.7.3.1 Prepare a Method Blank using 5.0 g of glass beads and 5 mL methanol. Add 5 µL of parent surrogate to the vial.

11.7.3.2 Prepare a LCS using 5.0 g glass beads, 5 mL of methanol , 5 µL of parent surrogate and 10 µL of spiking solution.

11.7.3.3 For the LCS and MS/MSD, add 100 µL medium level spike mix.

11.7.4 Using a vortex mixer, agitate sample for at least half a minute.

11.8 **Methanol Extraction of Soils (superceded 5030 method)**

11.8.1 See SOP ST-QA-0038 for the procedure for sub sampling.

11.8.2 Weigh 5 ± 0.05 g of the sample into a pre-weighed 40 mL glass labeled vial.

11.8.2.1 If the sample is suspected or known to have high concentrations of analytes, reduce the sample aliquot to 1.0 g

11.8.3 Record the weight in prep log (Attachment 1).

11.8.4 Seal the vial.

11.8.5 Add 5 mL of purge and trap methanol.

11.8.6 Add 5 µL medium level surrogate to each sample and QC samples.

11.8.6.1 Prepare a Method Blank using 5.0 g of glass beads and 5 mL methanol. Add 5µL of surrogate to the vial.

11.8.6.2 Prepare a LCS using 5.0 g glass beads, 5 mL methanol, 5 µL of surrogate and 20 µL of spiking solution.

11.8.6.3 For the MS/MSD, add 100 µL medium level spike mix.

11.8.7 Using a vortex mixer, agitate sample for at least half a minute.

11.9 **Volatile Analysis**:

11.9.1 Load each 40 mL VOA sample vial (and QC) in the purge and trap autosampler.

11.9.1.1 Medium Level Analysis

11.9.1.1.1 Rinse a glass-tight syringe with organic free water and fill the syringe with water. Bring volume to 5 mL add 5 µL of internal standards.

11.9.1.1.2 Add sample methanol extract to the syringe (no more than 100 µL for a 5 mL purge).

11.9.1.1.3 If less than 1 µL of methanol extract is to be added to the water, dilute the methanol extract using a serial dilution.

11.9.1.1.4 Transfer methanol extract/HPLC water in syringe to a labeled 40 mL VOA vial.

11.9.2 Record autosampler sample analysis sequence in logbook.

11.9.3 Start analysis.

11.9.4 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition.

11.9.4.1 For method 524.2 the desorb time should be a minimum of 2 minutes.

11.9.5 After desorption, bake the trap for 5 – 10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.9.6 When the standards and extracts are not being used, refrigerate them at 4 ± 2 ^{o}C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica ST-QAM.

12.2 Internal Standards

12.2.1 Samples that exhibit low IS recoveries < 50% or high IS recoveries > 100% are reanalyzed once to confirm matrix effect.

12.3 Manual Integrations

12.3.1 Identified compounds are reviewed for proper integration. Integrations are performed automatically by the data system. If necessary, manual integrations are performed and are documented by the analyst. Manual integrations are denoted with an “M” flag on the Target quantitation report. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices

12.4 Qualitative identification

12.4.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification:

12.4.1.1 Elution of sample component at the same GC retention time as the standard component; and

12.4.1.2 Correspondence of the sample component and the standard component characteristic ions.

12.4.1.2.1Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.4.2 The sample component retention time must compare to within ± 0.2 minutes. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.4.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.4.4 The relative intensities of ions should agree to within ± 30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance should be between 20 and 80%.)

12.4.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.5 Retention time criteria for samples

12.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

12.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

12.6 Tentatively Identified Compounds (TICs)

12.6.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.6.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.6.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%).

12.6.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.6.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

12.6.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.6.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client.

12.6.1.7 The first 20 TICs will be identified in a sample, unless a different number is specified by the client. See client requirement sheet.

12.7 Dilutions

12.7.1 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.

12.7.2 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.

12.7.2.1 Aqueous samples requiring less than a 1:1000 dilution can be diluted directly using a 5 mL syringe

12.7.2.2 Aqueous samples requiring less than a 1:5000 dilution can be diluted directly using a 25 mL syringe

12.7.2.3 Low level soil samples may re-analyzed using a 1 g sample aliquot or utilizing the methanol extraction technique.

12.8 Carryover

12.8.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.8.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.8.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analyses, carryover may have occurred.

12.8.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred

12.8.1.3 If the chromatographic profile resembles the previous sample, the results are questionable.

12.8.1.3.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Steps taken when an out of control situation occurs include demonstrating that all issues creating the out-of-control situation have been addressed, document the problem and the action taken to correct it, document that an in-control situation has been achieved.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 The method blank must have acceptable surrogate recoveries.

13.2.1.3 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.1.3.1 <u>Method Blank Contamination</u> – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.2.1.3.2 <u>Method Blank Surrogate excursion</u> – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-anlaysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.2.1.3.3 <u>For South Carolina compliance workthe Method Blank concentration must be below the RL</u>.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).

13.3.1.1 For long analyte spike list, marginal exceedances (ME) are allowed as follows:

13.3.1.2 less than 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.

13.3.1.3 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.

13.3.1.4 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.

13.3.1.5 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.

13.3.1.6 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.

13.3.1.7 More than 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.

13.3.1.8 No LCS recoveries may be outside the Marginal Exceedance limit.

13.3.1.9 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

13.3.1.10 Marginal exceedance is not allowed by all programs. See specific Project/Client CRM for details. The use of marginal exceedances is not allowed for South Carolina compliance samples. LCS recoveries must be within 70 -130% for South Carolina compliance work.

13.3.2 The LCS should have acceptable surrogate recoveries.

13.3.3 Corrective Action for LCS not meeting acceptance criteria:

13.3.3.3.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. . In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

13.3.3.3.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.3.3.3.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.

13.3.3.3.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

13.5 Sample result evaluation

13.5.1 Dilutions

13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.5.1.2 Dilution: Sample– An NCM is created when dilutions are required.

13.5.1.3 Dilution: Surrogate(s)/spikes diluted out– An NCM is generated to document the surrogates/spikes being diluted out.

13.5.2 Carryover

13.5.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.5.2.2 If a sample analyzed after a sample with high concentrations is non-detect for the high concentration analyted, carryover did not occur.

13.5.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.5.3 Internal Standards

13.5.3.1 Acceptance Criteria:

13.5.3.1.1 If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence.

13.5.3.1.2 If the EICP area for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified. (Some programs may

require that the midpoint of the initial calibration be used for ISTD monitoring. See the project CRM for specifics.)

13.5.3.2 Corrective Action for Internal Standards not meeting acceptance criteria:

13.5.3.2.1 Internal Standard excursion – high – High ISTD recovery indicates a potential low bias to analytical results. Instrument maintenance, if required, is done and affected samples are reanalyzed. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.

13.5.3.3 Internal Standard excursion – low – Low ISTD recovery indicates the potential for a high bias to analytical results. Samples that are non-detect for affected analytes may be reported with an NCM. Samples with positive hits above the RL for analytes associated with the poor ISTD recovery require re-analysis. Instrument maintenance, if required, is done. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.

13.6 Surrogate

13.6.1 All Surrogates should be within established control limits for accuracy (%Recovery).

13.6.2 Corrective Action for Surrogate not meeting acceptance criteria:

13.6.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.7 Insufficient Sample

13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, an NCM is created and a narrative comment stating such is included in the report's Case Narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for

pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".

16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8000B, 8000C, 8260B and 8260C

17.2 40CFR Part 136: "Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", Code of Federal Regulations, Revised July1, 1995, Method 624

17.3 USEPA Drinking Water method 524.2

17.4 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (ST-HS-0002), current revisions

17.6 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices

13.8 TestAmerica Policy CA-T-P-002, Selection of Calibration Points

17.7 Associated SOPs

17.7.1 ST-OP-0001, Organic Labware Cleaning Procedure
17.7.2 ST-PM-0002, Sample Receipt and Chain of Custody
17.7.3 ST-QA-0002, Standard and Reagent Preparation
17.7.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
17.7.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.7.6 ST-QA-0016, IDL/MDL Determination
17.7.7 ST-QA-0031, VOA Holding Blank Analysis
17.7.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS OF PREVIOUS REFERENCE METHOD

18.1 <u>Modification to Reference Method 624</u>

18.1.1 Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

18.1.2 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

18.1.3 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

18.1.4 Method 624 Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the Supelco K trap.

18.2 Modifications required for drinking water analysis by method 524.2

18.2.1 The lab analyzes groundwater by method 524.2 for specific client(s); a narrative is to be included in the applicable data packages that details deviations from the method.

18.2.2 Only one internal standard — Fluorobenzene — is required for this method. The lab uses 3 ISTDs.

18.2.3 The same analysis run may be used to satisfy the requirements for an LCS (also known as a laboratory fortified blank, LFB) and a continuing calibration verification sample. The LCS/CCV does not need to be a second source standard.

18.2.4 524.2 Section 7.1 of the source method requires that the trap packing materials be Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely uses Supleco K trap

18.2.5 524.2 Section 7.8.2 of the source method requires that each calibration standard be prepared by diluting the appropriate volume of the working standard with organic-free water adjusted to pH < 2 in a volumetric flask. TestAmerica prepares calibration standards by diluting the appropriate volume of the working standard with organic-free water at neutral pH.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Section 9.1.5: Blank, LCS & MS/MSD must be run in same clock for non-medium level samples

19.2 Section 10.3.2: The number of calibration points required for curves is clarified

19.3 Section 10.3.6.6: notes added to address client requirements and y-intercept

19.4 Section 10.3.7.9.1: added six point requirement for all quadratic curves

19.5 Section 10.4.4.2: Added corrective action for ICV failure (use of third standard)

19.6 Section 10.3.7.9.3: added note addressing client specific requirements

19.7 Section 12.7.1.1: added CLP allowance for reporting data within 10% of upper standard without dilution

19.8 Table 2: added allowance of other published BFB Tune criteria (i.e. EPA CLP)

19.9 Added Attachment 1 – example page from VOA Medium Level Extraction log.

19.10 Revision 17:

19.10.1 Fixed grammatical errors throughout SOP

19.10.2 Updated section 9.1 regarding MS/MSD duplicates being performed for separate QC codes.

19.10.3 Updated section 10.5.7 regarding CCV criteria testing.

19.10.4 Removed section 10.5.8.3 regarding passing CCV percentage calibration points in the initial calibration curve.

19.10.5 Updated section 11.7 and 11.8 regarding volumes of medium level surrogate and medium level spike mix used.

19.11 Rev 18:

19.11.1 Added information for Select Ion Monitoring Procedure to Sections 2, 3 and Appendix 1

19.12 Rev 19:

19.12.1 Section 3, updated SIM definition

19.12.2 Clarified Section 9.3 stating that the LCS and CCV can be from the same source

19.12.3 References to QuantIMS and Clouseau were removed and replaced with “LIMS”

19.12.4 Section 12, updated Corp. SOP reference

19.12.5 Section 12.7.1.1: removed the CLP allowance for reporting data within 10% of upper standard without dilution

19.12.6 Section 13 was re-written to include specific corrective action scenarios.

19.12.7 Section 17, updated Corp. SOP reference

19.12.8 Formatting and grammatical corrections

19.13 Rev 20:

19.13.1 Section 6.10.5: indicated where instrument run conditions can be found
19.13.2 Section 7.4: Updated Standards Log to Reagents in TALS nomenclature
19.13.3 Section 10.3: updated analysis volumes used
19.13.4 Section 10.3: Added that non-linear regressions can be forced through origin
19.13.5 Section 10.3.8: Corrected RSD criteria for EPA 624 method
19.13.6 Section 11.1: Updated screening procedures.
19.13.7 Section 11.4: Added 25mL method
19.13.8 Section 11.5: Corrected various omissions from prep instructions
19.13.9 Section 18.2: referenced procedure for notification of client of deviation from EPA method.

19.14 Revsion 21 (9/18/14):

19.14.1 Section 10.3.7 updated to note that quadratic regression curves are not allowed for South Carolina compliance samples
19.14.2 Section 13.3 updated to note that marginal exceedances are not allowed for South Carolina compliance work

19.15 Revsion 22 (11/3/14)

19.15.1 SOP combined with ST-MS-0002SC to document all requirements for South Carolina DHEC work.
19.15.2 Added Appendix 2 to contain standard and reagent information.

19.16 Revision 23 (12/1/14)

19.16.1 re-worded Section 2.4 to include Scan mode for 1,4-dioxane
19.16.2 added column specifics to Section 6
19.16.3 added Ottawa sand to Sections 7 and 9
19.16.4 Update Section 10 to disallow the use of "Grand Mean" for South Carolina Compliance work
19.16.5 Update %D info in Section 10.5
19.16.6 Updated Section 13 to include the 70 – 130% LCS recovery required for South Carolina compliance work.
19.16.7 Added Appendices with standard prep and concentration information

Table 1
Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Fluorobenzene	25	96	96
Chlorobenzene-d5	25	117	117
1,4-Dichlorobenzene-d4	25	152	152

Notes:
1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
2) Surrogate and internal standards may be combined in one solution.

Surrogate and Spike concentrations are listed on the Structure and Analysis Codes, which are in the attachment to this SOP.

Table 2
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

- BFB tuning criteria for mass 75 are 30 – 80% of mass 95 for method 524.2
- Alternatively, other documented tuning criteria (e.g. EPA CLP) may be used provided method performance is not aversely affected

Table 3
Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	67*	102*	65
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101*	103*	66
1,1-Dichloroethene	96	61	63, 98
Acrolein	56	55	58

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Iodomethane	142	127	141
Carbon disulfide	76	78	—
Trichlorotrifluoroethane	151	101	153
Acetone	58	43	—
Methylene chloride	84	86	49
tert-Butyl alcohol	59	74	—
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73	57	—
Hexane	57	43	—
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43*	72*	—
Tetrahydrofuran	71	42	72
Chloroform	83	85	47
1,2-Dichloroethane	62	98	64
Dibromomethane	93	95	172, 174, 176
1,4-Dioxane	88	58	43, 57
Vinyl acetate	43	86	—
1,1,1-Trichloroethane	97	99	117*
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130*	95*	132
Methylcyclohexane	55	83	98
1,2-Dichloropropane	63	41*	—
Bromodichloromethane	83	85	129*
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97*	83*	85
Chlorodibromomethane	129	127*	131*
Bromoform	173	171*	175*, 254
1,2,3-Trichloropropane	110*	77	75
Toluene-d_8 (Surrogate)	98	100	—
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91*	92*	65
4-Methyl-2-pentanone	43*	58*	85, 100
Tetrachloroethene	164	129	131, 166
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Ethylbenzene	91	106	—
Xylenes	106	91	—
Styrene	104	78	103
Dibromofluoromethane	113	111	192
Dichlorobenzene (all isomers)	146	111	148
trans 1,4-Dichloro-2-butene	53	88	75
1,1,2,2-Tetrachloroethane	83	85	131,133
Allyl Chloride	76	41	39, 78
Acetonitrile	41	40	39
Dichlorofluoromethane	67	69	—
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42, 43
Propionitrile	54	52	55
Methacrylonitrile	41	52	39
Isobutanol	43	42	74
Methyl methacrylate	41	69	39
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	—
Ethyl Acetate	43*	61*	88*
2-Nitropropane	46	43	—
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	77
1,2-Dichlorobenzene	111	146	148
1,3-Dichlorobenzene	111	146	148
1,4-Dichlorobenzene	111	146	148
Nonanol	57	98	41
t-Butyl alcohol	59	41	—
1-Chlorohexane	91	55	43
Ethanol	45	46	—
TAME	73	87	43
ETBE	59	87	41
DIPE (Diisopropyl ether)	45	87	59
2,2-Dimethylpentane	45	46	—
2,4-Dimethylpentane	57	85	43
2,2,3-Trimethylbutane	57	43	85
3,3-Dimethylpentane	43	71	85
2-Methylhexane	43	57	85
2,3-Dimethylpentane	56	71	43
3-Methylhexane	43	57	71

Characteristic ions

Compound	Primary*	Secondary	Tertiary
3-Ethylpentane	43	71	55
Heptane	43	57	71
Dimethylsulfide	94	79	45
1,3,5-Trichlorbenzene	180	182	145

The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.
*Primary/secondary and/or tertiary ions are switched from order in Method based on signal intensity and co-elutions.

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

TestAmerica St. Louis has established a default minimum response factor of 0.01 for compounds not identified in this table, except for Acrolein, Acetonitrile, Isobutanol, Tetrahydrofuran, Propionitrile, n-butanol, 1,4-Dioxane, 2-chloroethyl vinyl ether, Cyclohexanone, nonanol (25 mL purge) and Acrolein, Acetonitrile, Tetrahydrofuran,

Propionitrile, Isobutanoln-butanol, 1,4-Dioxane, Cyclohexanone (5 mL purge), which have a minimum response factor of 0.001.

Table 5
Assigned Surrogates/Internal Standards for Instruments using Instrument MSG

Compound	Assigned Surrogate	Assigned Internal Standard
Dichlorodifluoromethane	Dibromofluoromethane	Fluorobenzene
Freon-114	Dibromofluoromethane	Fluorobenzene
Chloromethane	Dibromofluoromethane	Fluorobenzene
Vinyl Chloride	Dibromofluoromethane	Fluorobenzene
Bromomethane	Dibromofluoromethane	Fluorobenzene
Chloroethane	Dibromofluoromethane	Fluorobenzene
Trichlorofluoromethane	Dibromofluoromethane	Fluorobenzene
Diethyl Ether	Dibromofluoromethane	Fluorobenzene
1,1,2-Trichlorofluoroethane	Dibromofluoromethane	Fluorobenzene
Acrolein	Dibromofluoromethane	Fluorobenzene
Acetone	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethene	Dibromofluoromethane	Fluorobenzene
Acetonitrile	Dibromofluoromethane	Fluorobenzene
Iodomethane	Dibromofluoromethane	Fluorobenzene
Methyl Acetate	Dibromofluoromethane	Fluorobenzene
Allyl chloride	Dibromofluoromethane	Fluorobenzene
Carbon Disulfide	Dibromofluoromethane	Fluorobenzene
Methylene Chloride	Dibromofluoromethane	Fluorobenzene
Acrylonitrile	Dibromofluoromethane	Fluorobenzene
MTBE	Dibromofluoromethane	Fluorobenzene
trans-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
n-Hexane	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethane	Dibromofluoromethane	Fluorobenzene
1,2-Dichloroethene (total)	Dibromofluoromethane	Fluorobenzene
Vinyl acetate	Dibromofluoromethane	Fluorobenzene
2-Chloro-1,3-butadiene	Dibromofluoromethane	Fluorobenzene
2-Butoxyethanol	Dibromofluoromethane	Fluorobenzene
2-Butanone	Dibromofluoromethane	Fluorobenzene
Propionitrile	Dibromofluoromethane	Fluorobenzene
2,2-Dichloropropane	Dibromofluoromethane	Fluorobenzene
cis-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
Isobutanol	Dibromofluoromethane	Fluorobenzene
Ethyl Acetate	Dibromofluoromethane	Fluorobenzene
Methacrylonitrile	Dibromofluoromethane	Fluorobenzene

Compound	Assigned Surrogate	Assigned Internal Standard
Chloroform	Dibromofluoromethane	Fluorobenzene
Bromochloromethane	Dibromofluoromethane	Fluorobenzene
Tetrahydrofuran	Dibromofluoromethane	Fluorobenzene
n-butanol	1,2-Dichloroethane-d4	Fluorobenzene
1,1,1-Trichloroethane	1,2-Dichloroethane-d4	Fluorobenzene
Cyclohexane	1,2-Dichloroethane-d4	Fluorobenzene
1,1-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
Carbon Tetrachloride	1,2-Dichloroethane-d4	Fluorobenzene
Heptane	1,2-Dichloroethane-d4	Fluorobenzene
Benzene	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloroethane	1,2-Dichloroethane-d4	Fluorobenzene
Trichloroethene	1,2-Dichloroethane-d4	Fluorobenzene
Methyl cyclohexane	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloropropane	1,2-Dichloroethane-d4	Fluorobenzene
Methyl methacrylate	1,2-Dichloroethane-d4	Fluorobenzene
Bromodichloromethane	1,2-Dichloroethane-d4	Fluorobenzene
Dibromomethane	1,2-Dichloroethane-d4	Fluorobenzene
1,4-Dioxane	1,2-Dichloroethane-d4	Fluorobenzene
4-Methyl-2-pentanone (MIBK)	1,2-Dichloroethane-d4	Fluorobenzene
2-Chloroethyl vinyl ether	1,2-Dichloroethane-d4	Fluorobenzene
Cis-1,3-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
Dimethyl disulfide	1,2-Dichloroethane-d4	Fluorobenzene
2-Nitropropane	1,2-Dichloroethane-d4	Fluorobenzene
Toluene	Toluene-d8	Chlorobenzene-d5
trans-1,3-Dichloropropene	Toluene-d8	Chlorobenzene-d5
Ethyl methacrylate	Toluene-d8	Chlorobenzene-d5
1,1,2-Trichloroethane	Toluene-d8	Chlorobenzene-d5
2-Hexanone	Toluene-d8	Chlorobenzene-d5
1,3-Dichloropropane	Toluene-d8	Chlorobenzene-d5
Tetrachloroethene	Toluene-d8	Chlorobenzene-d5
Chlorodibromomethane	Toluene-d8	Chlorobenzene-d5
1,2-Dibromoethane	Toluene-d8	Chlorobenzene-d5
Chlorobenzene	Toluene-d8	Chlorobenzene-d5
1,1,1,2-Tetrachloroethane	Toluene-d8	Chlorobenzene-d5
Ethylbenzene	Toluene-d8	Chlorobenzene-d5
m,p-Xylenes	Toluene-d8	Chlorobenzene-d5
o-Xylenes	Toluene-d8	Chlorobenzene-d5
Styrene	Toluene-d8	Chlorobenzene-d5
Bromoform	Toluene-d8	Chlorobenzene-d5
Isopropylbenzene	Toluene-d8	Chlorobenzene-d5

Compound	Assigned Surrogate	Assigned Internal Standard
Cyclohexanone	Toluene-d8	Chlorobenzene-d5
1,1,2,2-Tetrachloroethane	Toluene-d8	Chlorobenzene-d5
1,2,3-Trichloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Bromobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
n-Propylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
trans-1,4-dichlorobenzene-2-butene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
2-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
t-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Pentachloroethane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
sec-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Isopropyltoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,4-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
n-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Nonanal	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dibromo-3-chloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Hexachlorobutadiene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Naphthalene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,3-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorophenyl methyl sulfide	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4

Assigned Surrogates/Internal Standards for Instruments using Instruments MSF, MSL, MSM, MSN

Compound	Assigned Surrogate	Assigned Internal Standard
Dichlorodifluoromethane	Dibromofluoromethane	Fluorobenzene
Freon-114	Dibromofluoromethane	Fluorobenzene
Chloromethane	Dibromofluoromethane	Fluorobenzene
Vinyl Chloride	Dibromofluoromethane	Fluorobenzene
Bromomethane	Dibromofluoromethane	Fluorobenzene

Compound	Assigned Surrogate	Assigned Internal Standard
Chloroethane	Dibromofluoromethane	Fluorobenzene
Trichlorofluoromethane	Dibromofluoromethane	Fluorobenzene
Diethyl Ether	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethene	Dibromofluoromethane	Fluorobenzene
1,1,2-Trichlorofluoroethane	Dibromofluoromethane	Fluorobenzene
Carbon Disulfide	Dibromofluoromethane	Fluorobenzene
Iodomethane	Dibromofluoromethane	Fluorobenzene
Acrolein	Dibromofluoromethane	Fluorobenzene
Allyl chloride	Dibromofluoromethane	Fluorobenzene
Methylene Chloride	Dibromofluoromethane	Fluorobenzene
Acetone	Dibromofluoromethane	Fluorobenzene
Methyl Acetate	Dibromofluoromethane	Fluorobenzene
trans-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
n-Hexane	Dibromofluoromethane	Fluorobenzene
Acetonitrile	Dibromofluoromethane	Fluorobenzene
MTBE	Dibromofluoromethane	Fluorobenzene
2-Chloro-1,3-butadiene	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethane	Dibromofluoromethane	Fluorobenzene
1,2-Dichloroethene (total)	Dibromofluoromethane	Fluorobenzene
Acrylonitrile	Dibromofluoromethane	Fluorobenzene
Vinyl acetate	Dibromofluoromethane	Fluorobenzene
cis-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
2,2-Dichloropropane	Dibromofluoromethane	Fluorobenzene
Bromochloromethane	Dibromofluoromethane	Fluorobenzene
2-Butoxyethanol	Dibromofluoromethane	Fluorobenzene
Cyclohexane	Dibromofluoromethane	Fluorobenzene
Chloroform	Dibromofluoromethane	Fluorobenzene
t-Butyl Alcohol	Dibromofluoromethane	Fluorobenzene
Diisopropyl Ether	Dibromofluoromethane	Fluorobenzene
ETBE	Dibromofluoromethane	Fluorobenzene
Ethanol	Dibromofluoromethane	Fluorobenzene
2,2-Dimethylpentane	Dibromofluoromethane	Fluorobenzene
2,4-Dimethylpentane	Dibromofluoromethane	Fluorobenzene
2,2,3-Trimethylbutane	Dibromofluoromethane	Fluorobenzene
3,3-Dimethylpentane	Dibromofluoromethane	Fluorobenzene
Ethyl Acetate	Dibromofluoromethane	Fluorobenzene
Carbon Tetrachloride	Dibromofluoromethane	Fluorobenzene
Tetrahydrofuran	Dibromofluoromethane	Fluorobenzene
1,1,1-Trichloroethane	1,2-Dichloroethane-d4	Fluorobenzene

Compound	Assigned Surrogate	Assigned Internal Standard
2-Butanone	1,2-Dichloroethane-d4	Fluorobenzene
1,1-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
Heptane	1,2-Dichloroethane-d4	Fluorobenzene
Benzene	1,2-Dichloroethane-d4	Fluorobenzene
Propionitrile	1,2-Dichloroethane-d4	Fluorobenzene
Methacrylonitrile	1,2-Dichloroethane-d4	Fluorobenzene
Isobutanol	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloroethane	1,2-Dichloroethane-d4	Fluorobenzene
Trichloroethene	1,2-Dichloroethane-d4	Fluorobenzene
Methyl cyclohexane	1,2-Dichloroethane-d4	Fluorobenzene
n-butanol	1,2-Dichloroethane-d4	Fluorobenzene
Dibromomethane	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloropropane	1,2-Dichloroethane-d4	Fluorobenzene
Bromodichloromethane	1,2-Dichloroethane-d4	Fluorobenzene
Methyl methacrylate	1,2-Dichloroethane-d4	Fluorobenzene
1,4-Dioxane	1,2-Dichloroethane-d4	Fluorobenzene
Cis-1,3-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
2-Chloroethylvinyl ether	1,2-Dichloroethane-d4	Fluorobenzene
TAME	1,2-Dichloroethane-d4	Fluorobenzene
2-Methylhexane	1,2-Dichloroethane-d4	Fluorobenzene
2,3-Dimethylpentane	1,2-Dichloroethane-d4	Fluorobenzene
3-Methylhexane	1,2-Dichloroethane-d4	Fluorobenzene
3-Ethypentane	1,2-Dichloroethane-d4	Fluorobenzene
Heptane	1,2-Dichloroethane-d4	Fluorobenzene
Toluene	Toluene-d8	Chlorobenzene-d5
Dimethyl Disulfide	Toluene-d8	Chlorobenzene-d5
2-Nitropropane	Toluene-d8	Chlorobenzene-d5
4-Methyl-2-pentanone (MEK)	Toluene-d8	Chlorobenzene-d5
trans-1,3-Dichloropropene	Toluene-d8	Chlorobenzene-d5
Tetrachloroethene	Toluene-d8	Chlorobenzene-d5
Ethyl methacrylate	Toluene-d8	Chlorobenzene-d5
1,1,2-Trichloroethane	Toluene-d8	Chlorobenzene-d5
Chlorodibromomethane	Toluene-d8	Chlorobenzene-d5
1,3-Dichloropropane	Toluene-d8	Chlorobenzene-d5
1,2-Dibromoethane	Toluene-d8	Chlorobenzene-d5
2-Hexanone	Toluene-d8	Chlorobenzene-d5
Ethylbenzene	Toluene-d8	Chlorobenzene-d5
Chlorobenzene	Toluene-d8	Chlorobenzene-d5
1,1,1,2-Tetrachloroethane	Toluene-d8	Chlorobenzene-d5

Compound	Assigned Surrogate	Assigned Internal Standard
m,p-Xylenes	Toluene-d8	Chlorobenzene-d5
o-Xylenes	Toluene-d8	Chlorobenzene-d5
Styrene	Toluene-d8	Chlorobenzene-d5
1-Chlorohexane	Toluene-d8	Chlorobenzene-d5
Bromoform	Toluene-d8	1,4-Dichlorobenzene-d4
Isopropylbenzene	Toluene-d8	1,4-Dichlorobenzene-d4
n-Propylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,1,2,2-Tetrachloroethane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Bromobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
2-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
trans-1,4-dichlorobenzene-2-butene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,3-Trichloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Cyclohexanone	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
t-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Pentachloroethane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
sec-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Isopropyltoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,4-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
n-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dibromo-3-chloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Hexachlorobutadiene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Naphthalene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,3-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorophenyl methyl sulfide	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4

Attachment 1
Medium Level Soil Extraction Log

Analyst: _____ Extraction Date: _____

Methanol Lot ID: _____ Batch # _____

Balance ID: _____

The final volume of all extracts is 5 mL of methanol.
All weights are expressed in grams.

Sample ID	Sample Weight	Methanol volume	Sample spiked	Spike verification

QC Standards: LCS/MS/MSD_____

Surrogate_____

Appendix 1

1,4-Dioxane 8260B and 8260C SIM Method

Standards

Internal Standard Working Mix 1,4-Dioxane-d8 50 ppm

Calibration Standard Working Mix 1,4-Dioxane 50 ppm

8260 IS Working Mix 8260 IS 1 ppm

Calibration Table

Level	1,4-Diox. Mix in 5ml	1,4-Diox. Mix in 20ml	d8-1,4-Diox in 20ml	8260 IS Mix in 20ml
5 ppb	0.5 µL	2µL	40 µL	10 µL
10 ppb	1.0 µL	4µL	40 µL	10 µL
20 ppb	2.0 µL	8µL	40 µL	10 µL
50 ppb	5.0 µL	20µL	40 µL	10 µL
100 ppb	10 µL	40 µL	40 µL	10 µL
200 ppb	20 µL	80 µL	40 µL	10 µL

d8-1,4-Dioxane IS concentration in sample 100 ppb

1,4-Dioxane ICV/LCS/MS/MSD concentration 20 ppb

8260 IS concentration in samples 0.5 ppb

Need MDL verification after BFB of each clock 2 ppb (based on current MDL 0.74 ppb)

Spike d8-1,4-Dioxane **and** 8260 IS in all analyses

GC/MS Method – 8260SIM Concentrator Method – 8260SIM

APPENDIX 2

Standards Prep information:

WM=Working Mix (single mix of all calibrated analytes at concentration levels as indicated in Appendix 3

Surr=Surrogate mix (single mix of all applicable surrogates at concentration levels as indicated in Appendix 3

8260 (B & C) - all matrices)

All calibration levels are calculated from 5mL of the appropriate matrix. All calibration levels are spiked with 50ug/L Internal Standard mix.

Level 1: 0.2uL WM and 0.2uL Surr

Level 2: 0.5uL WM and 0.5uL Surr

Level 3: 1uL WM and 1uL Surr

Level 4: 2uL WM and 2uL Surr

Level 5: 4uL WM and 4uL Surr

Level 6: 10uL WM and 10uL Surr

Level 7: 20uL WM and 20uL Surr

Level 8: 40uL WM and 40uL Surr

8260 (B & C) Low Level

All calibration levels are calculated from 25mL of water. All calibration levels are spiked with 10ug/L Internal Standard mix.

Level 1: 0.5uL WM and 0.5uL Surr

Level 2: 1uL WM and 1uL Surr

Level 3: 2uL WM and 2uL Surr

Level 4: 4uL WM and 4uL Surr

Level 5: 10uL WM and 10uL Surr

Level 6: 20uL WM and 20uL Surr

Level 7: 40uL WM and 40uL Surr

APPENDIX 3

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8260MegaMixSS_00020

Reagent ID: 8260MegaMixSS_00020

Type:	ASTD	Expiration Date:	02/29/2016
Description:	8260 List 1/Std #1 MegaMix Second Source	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storag	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A093733
Creation Date:	10/29/2014	Vendor Cat #:	567641.SEC
Container(s):	489974		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,1,1,2-Tetrachloroethane					2000.00000	ug/mL
1,1,1-Trichloroethane					2000.00000	ug/mL
1,1,2,2-Tetrachloroethane					2000.00000	ug/mL
1,1,2-Trichloro-1,2,2-trifluoroethane					2000.00000	ug/mL
1,1,2-Trichloroethane					2000.00000	ug/mL
1,1-Dichloroethane					2000.00000	ug/mL
1,1-Dichloroethene					2000.00000	ug/mL
1,1-Dichloropropene					2000.00000	ug/mL
1,2,3-Trichlorobenzene					2000.00000	ug/mL
1,2,3-Trichloropropane					2000.00000	ug/mL
1,2,4-Trichlorobenzene					2000.00000	ug/mL
1,2,4-Trimethylbenzene					2000.00000	ug/mL
1,2-Dibromo-3-Chloropropane					2000.00000	ug/mL
1,2-Dichlorobenzene					2000.00000	ug/mL
1,2-Dichloroethane					2000.00000	ug/mL
1,2-Dichloropropane					2000.00000	ug/mL
1,3,5-Trimethylbenzene					2000.00000	ug/mL
1,3-Dichlorobenzene					2000.00000	ug/mL
1,3-Dichloropropane					2000.00000	ug/mL
1,4-Dichlorobenzene					2000.00000	ug/mL
1,4-Dioxane					40000.00000	ug/mL
2,2-Dichloropropane					2000.00000	ug/mL
2-Chlorotoluene					2000.00000	ug/mL
2-Methyl-2-propanol					20000.00000	ug/mL
3-Chloro-1-propene					2000.00000	ug/mL
4-Chlorotoluene					2000.00000	ug/mL
4-Isopropyltoluene					2000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8260MegaMixSS_00020

Reagent ID: 8260MegaMixSS_00020

Type:	ASTD	Expiration Date:	02/29/2016
Description:	8260 List 1/Std #1 MegaMix Second Source	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storag	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A093733
Creation Date:	10/29/2014	Vendor Cat #:	567641.SEC
Container(s):	489974		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Acrylonitrile					20000.00000	ug/mL
Benzene					2000.00000	ug/mL
Bromobenzene					2000.00000	ug/mL
Bromoform					2000.00000	ug/mL
Carbon disulfide					2000.00000	ug/mL
Carbon tetrachloride					2000.00000	ug/mL
Chlorobenzene					2000.00000	ug/mL
Chlorobromomethane					2000.00000	ug/mL
Chlorodibromomethane					2000.00000	ug/mL
Chloroform					2000.00000	ug/mL
cis-1,2-Dichloroethene					2000.00000	ug/mL
cis-1,3-Dichloropropene					2000.00000	ug/mL
Cyclohexane					2000.00000	ug/mL
Dibromomethane					2000.00000	ug/mL
Dichlorobromomethane					2000.00000	ug/mL
Ethyl ether					2000.00000	ug/mL
Ethyl methacrylate					2000.00000	ug/mL
Ethylbenzene					2000.00000	ug/mL
Ethylene Dibromide					2000.00000	ug/mL
Hexachlorobutadiene					2000.00000	ug/mL
Hexane					2000.00000	ug/mL
Iodomethane					2000.00000	ug/mL
Isobutyl alcohol					50000.00000	ug/mL
Isopropylbenzene					2000.00000	ug/mL
Methyl acetate					10000.00000	ug/mL
Methyl tert-butyl ether					2000.00000	ug/mL
Methylcyclohexane					2000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8260MegaMixSS_00020

Reagent ID: 8260MegaMixSS_00020

Type:	ASTD	Expiration Date:	02/29/2016
Description:	8260 List 1/Std #1 MegaMix Second Source	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storag	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A093733
Creation Date:	10/29/2014	Vendor Cat #:	567641.SEC
Container(s):	489974		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Methylene Chloride					2000.00000	ug/mL
m-Xylene & p-Xylene					2000.00000	ug/mL
Naphthalene					2000.00000	ug/mL
n-Butylbenzene					2000.00000	ug/mL
n-Heptane					2000.00000	ug/mL
N-Propylbenzene					2000.00000	ug/mL
o-Xylene					2000.00000	ug/mL
sec-Butylbenzene					2000.00000	ug/mL
Styrene					2000.00000	ug/mL
tert-Butylbenzene					2000.00000	ug/mL
Tetrachloroethene					2000.00000	ug/mL
Tetrahydrofuran					4000.00000	ug/mL
Toluene					2000.00000	ug/mL
trans-1,2-Dichloroethene					2000.00000	ug/mL
trans-1,3-Dichloropropene					2000.00000	ug/mL
trans-1,4-Dichloro-2-butene					2000.00000	ug/mL
Trichloroethene					2000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8260 IS(2014)_00011

Reagent ID: 8260 IS(2014)_00011

Type:	ASTD	Expiration Date:	05/31/2019
Description:	8260 IS Mix (3-Comp.) 2500ppm	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0103459
Creation Date:	09/06/2014	Vendor Cat #:	30241
Container(s):	444479		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,4-Dichlorobenzene-d4					2500.00000	ug/mL
Chlorobenzene-d5					2500.00000	ug/mL
Fluorobenzene					2500.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8260Gases_00078

Reagent ID: 8260Gases_00078

Type:	ASTD	Expiration Date:	09/30/2016
Description:	8260 List 1/Std #3 Gases	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storag	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0105755
Creation Date:	10/30/2014	Vendor Cat #:	567645
Container(s):	489981		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Bromomethane					2000.00000	ug/mL
Butadiene					2000.00000	ug/mL
Chloroethane					2000.00000	ug/mL
Chloromethane					2000.00000	ug/mL
Dichlorodifluoromethane					2000.00000	ug/mL
Dichlorofluoromethane					2000.00000	ug/mL
Trichlorofluoromethane					2000.00000	ug/mL
Vinyl chloride					2000.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

8260_Surr_00018

Reagent ID: 8260_Surr_00018

Type:	ASTD	Expiration Date:	05/31/2019
Description:	8260 Surrogate Std New Version	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Standards Storage	Vendor:	Restek
Reagent Volume:	5.000 mL	Vendor Lot #:	A0103672
Creation Date:	09/06/2014	Vendor Cat #:	567650
Container(s):	444473		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,2-Dichloroethane-d4 (Surr)					2500.00000	ug/mL
4-Bromofluorobenzene (Surr)					2500.00000	ug/mL
Dibromofluoromethane (Surr)					2500.00000	ug/mL
Toluene-d8 (Surr)					2500.00000	ug/mL

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Title: ANALYSIS OF METALS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY [SW-846 6020; SW-846 6020A; EPA 200.8]

Approvals (Signature/Date):

Karen Dickson 6/22/15 *For* Kristen Ely — Date Metals Supervisor	Elaine Wild FOR 6/22/15 Michael Ridenhower — Date Health & Safety Manager / Coordinator
Marti Ward 6.22.15 Marti Ward — Date Quality Assurance Manager	Elaine Wild 6/22/15 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-MT-0001 Rev. 23

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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA SW846 Method 6020, 6020A and EPA 200.8 .

1.2. This method is applicable to surface, and saline waters; soil and waste samples.

1.3. The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8) and the soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.

1.3.1. The Technetium-99 soil preparation procedure is found in SOP ST-RC-0125, Determination of Technetium-99 Using Eichrom TEVA Resin,

1.4. The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

1.4.1. Additional elements may be amendable to this method provided the laboratory has established a MDL and the elements meets the QC requirements as prescribed in the associated preparation and analysis SOP.

2.0 SUMMARY OF METHOD

2.1. Sample digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio frequency plasma. There the sample is decomposed and desolvated. The ions produces are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole or octupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3.0 DEFINITIONS

3.1. See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2. EPA and SW methodology use different terminology. Our SOP references the SW 846 terminology:

3.2.1. The ICV satisfies the QCS requirements found in method 200.8 .

3.2.2. The LCS satisfies the requirements of the LFB found in method 200.8.

3.2.3. The MS satisfies the requirements of the LFM found in method 200.8.

3.2.4. The MB satisfies the requirements of the LRB found in method 200.8.

3.3. Dissolved Metals: Those elements which pass through a 0.45 µm membrane filter (Sample is acidified after filtration)

3.4. Suspended Metals: Those elements retained by a 0.45 µm filter

3.5. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion

3.6. Dilution Test: the terminology “dilution test” found in later versions of 200.8 and 6020A is referred to as a Serial Dilution in this SOP.

4.0 INTERFERENCES

4.1. Isobaric elemental interferences: Isobaric elemental interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.

4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences. This may be done with either interference equations or collision cell technology.

4.3. Common molecular ion interferences are listed in Table 3 of this SOP.

4.4. Matrix interferences: Internal standards are used to correct for some matrix interferences.
4.4.1. Internal standards are added at a level to give approximately 100,000 - 10,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ± 50 amu of the mass of the affected analyte.
4.4.2. Severe matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the initial calibration blank.

5.0 SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
5.2.1. The ICP plasma emits strong UV light, harmful to vision. Analysts must avoid looking directly at the plasma.

5.3. PRIMARY MATERIALS USED
5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1. PerkinElmer® ELAN 6100/ PerkinElmer® ELAN 9000 /Agilent 7500/ Agilent 7700 (all with auto samplers)

6.2. Helium gas: 5.5 trace analytical grade

6.3. Argon gas: High-purity grade (99.99%)

6.4. Chiller (water cooling device)

6.5. Peristaltic Pump

6.6. Calibrated automatic pipettes

6.7. Teflon® flasks

6.8. Instrument software: ELAN version 2.3.2 / ELAN version 3.3 / Mass Hunter version B.01.01.

7.0 REAGENTS AND STANDARD

7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2. Concentrated nitric acid (HNO_3), trace metal grade

7.3. Concentrated hydrochloric acid (HCl), trace metal grade

7.4. DI water from the Millipore unit

7.4.1. Water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Water must be shown to have a resistivity greater than or equal to 16.67 Mohm-cm.

7.5. Standards

7.5.1. Purchased as custom multi-element mixes or as single-element solutions

7.5.2. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.

7.5.3. Working calibration and calibration verification solutions may be used for up to 1 week and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 2% hydrochloric and 2% nitric acid.

7.5.4. Internal Standard Solution: Prepare internal standards (Au, Sc, Ge, In, Ho, Li6, Ir) when needed.

7.5.5. Tuning solution: Prepare tuning solution (Be, Ba, Ce, Co, In, Pb, Li, Mg, Rh, Tl, Y) when needed.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure, ST-IP-0013.

8.3. Soil or waste samples are digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure, ST-IP-0002.

8.4. Digestate holding time is 6 months from sample collection.

9.0 QUALITY CONTROL

9.1. **Batch**

9.1.1. A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3. For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD an LCS Duplicate (LCSD) is prepared and analyzed.

9.2. **Method Blank (MB)**

9.2.1. A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2. A method blank must be prepared with every sample batch.

9.2.3. DI water is used for the method blank associated with water batches. 2% HNO_3 is used instead of DI for gold, tantalum and palladium water batches**.** DI and glass beads are used with solid batches.

9.2.4. See Section 13 for acceptance criteria.

9.3. **Laboratory Control Sample (LCS)**

9.3.1. An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2. An LCS must be prepared with every sample batch.

9.3.3. DI water, spiked with the analytes of interest, is used for the LCS associated with water batches. 2% HNO_3 is used instead of DI for gold, tantalum and palladium for water batches. A commercially available solid reference material is used for the LCS associated with solid batches. Where reference material is not available, spiked glass beads comprise the LCS.

9.3.4. See Section 13 for acceptance criteria.

9.4. **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1. A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2. See Section 13 for acceptance criteria.

9.5. **Serial Dilution**

9.5.1. A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.

9.5.2. The test is performed by running a sample at a 5x (1:4) dilution.

9.5.3. Samples identified as field blanks cannot be used for dilution tests.

9.5.4. See Section 13 for acceptance criteria.

9.6. **Post Digestion Spike** (PDS)

9.6.1. A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process, with a spike concentration between 10-100 times the MDL (unless specific project/program criteria is given)

9.6.2. 200.8: A PDS is not applicable for this method

9.6.3. 6020: A PDS is analyzed with every batch

9.6.4. 6020A: The method stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed.

9.6.5. For client project or programs requiring a PDS, the laboratory will include a PDS in the batch in addition to the serial dilution. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.
9.6.6. See Section 13 for acceptance criteria.

9.7. **Method of Standard Addition** (MSA)
9.7.1. **6020 and 6020A**
9.7.2. This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.
9.7.3. MSA are not required by methods 6020 or 6020A
9.7.4. MSA are not considered standard batch QC and if required by the client, must appear in the sample comment section in LIMS.
9.7.5. MSA is required by SW846 Method 1311 when the MS/MSD recovery is less than 50%, analyte concentration is less than and within 20% of its regulatory limit.

9.8. **Procedural Variations/ Nonconformance and Corrective Action**
9.8.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

Follow the instrument start-up procedure outlined in the manufacturers operating manual.
10.1. Cone Conditioning
10.1.1. Aspirating a 25% tap water / 75% ICPMS rinse solution for at least 1 hour can enhance instrument performance. This procedure should be used daily after a thorough cleaning of the interface cones or the installation of new cones takes place.

10.2. Rinse Time Determination
10.2.1. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in this SOP it can be demonstrated that a shorter rinse time may be used.
10.2.1.1. To determine the appropriate rinse time, a linear range verification standard should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for the system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

10.3. Instrument Tuning (Agilent)
10.3.1. Frequency:
10.3.1.1. Daily with each initial calibration
10.3.2. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Li, Y, Tl, Co, In, and Ce.
10.3.3. Tune Criteria:
10.3.3.1. Mass calibration and resolution checks must be documented and included as part of the raw data package.
10.3.3.1.1. Resolution
10.3.3.1.1.1 6020 and 6020A: peak width must be < 0.9 amu at 10% peak height
10.3.3.1.1.2 200.8: peak width of approximately 0.75 amu at 5% peak height

10.3.3.1.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.

10.3.3.1.3. The tuning elements must have RSD below 5%. Doubly-charged ions and oxides must be below 3.0%.

10.3.3.1.4. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.4. Instrument Tuning (Perkin Elmer)

10.4.1. Frequency:

10.4.1.1. Daily with each initial calibration

10.4.2. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Li, Mg, Rh. Ce, Pb and Ba.

10.4.3. Tune Criteria:

10.4.3.1. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.4.3.1.1. Resolution

10.4.3.1.1.1 6020 and 6020A: peak width must be < 0.9 amu at 10% peak height

10.4.3.1.1.2 200.8: peak width of approximately 0.75 amu at 5% peak height

10.4.3.1.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.

10.4.3.1.3. Using the Tuning Solution, an Auto-lens calibration is performed to ensure that optimum voltages are being applied to the Auto-lens. The default calibration should range from 4- 12 volts

10.4.3.1.4. Mg should be greater than 20,000 counts. Pb should be at or above 100,000 counts. Rh should be at or above 150,000. The background should be less than or equal to 30 counts. These are manufacturer recommendations, not requirements.

10.4.3.1.5. The tuning elements must have RSD below 5%. Doubly-charged ions and oxides must be below 3.0%.

10.4.3.1.6. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.5. **Initial Calibration**

10.5.1. Multi-point Calibration:

10.5.1.1. A calibration curve, consisting of 3 standards and a blank, must be analyzed daily.

10.5.1.2. Calibration criteria:

10.5.1.2.1. Correlation Coefficient of ≥ 0.998

10.5.1.2.2. The low level standard in the curve must be at or below the laboratory's routine reporting limit.

10.5.1.2.2.1 If a client requested reporting limit is below the laboratory's routine reporting limit and thus below the low level verification standard, the laboratory will discuss with the client, prior to sample analysis, how to proceed with this requirement.

10.6. **Initial Calibration Verification/Low Level Initial Calibration Verification/Initial Calibration Blank (ICV/LLICV/ICB)**

10.6.1. **ICV**

10.6.1.1. Secondary source, used to verify the initial calibration accuracy.

10.6.1.2. Frequency: Perform with each initial calibration
10.6.1.3. Criteria: ± 10%
10.6.1.4. Action upon failure:
10.6.1.4.1. If the ICV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
10.6.1.4.2. For all other non-conformances, the samples must be re-analyzed.

10.6.2. **LLICV**
10.6.2.1. Applicable to 6020A only
10.6.2.2. Same source as calibration
10.6.2.3. Frequency: Perform with each initial calibration.
10.6.2.4. Criteria: ± 30%
10.6.2.5. Action upon failure:
10.6.2.5.1. If the LLICV fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.6.2.5.2. If the LLICV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.6.2.5.3. For all other non-conformances, the samples must be re-analyzed.

10.6.3. **ICB**
10.6.3.1. Frequency: An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.
10.6.3.2. Criteria: The ICB result must fall within ± the RL from zero.
10.6.3.3. Action upon failure:
10.6.3.3.1. If the ICB fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.6.3.3.2. If the ICB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.6.3.3.3. For all other non-conformances, the samples must be re-analyzed.

10.6.4. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.
10.6.5. Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.7. **Continuing Calibration Verification/Low Level Continuing Calibration Verification/Continuing Calibration Blank (CCV/LLCCV/CCB)**
10.7.1. Calibration is monitored throughout the analytical run through the analysis of a known mid-level calibration standard.
10.7.2. **CCV**
10.7.2.1. A CCV may be a second source or the same source as the calibration.
10.7.2.2. Frequency: Analyte response factors must be verified at the beginning of each analytical

run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run.

10.7.2.3. Criteria:

10.7.2.3.1. For **200.8**: The CCV must fall within 15% of the true value for that solution.

10.7.2.3.2. For **6020 and 6020A:** The CCV must fall within 10% of the true value for that solution.

10.7.2.4. Action upon failure:

10.7.2.4.1. If the CCV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.7.2.4.2. For all other non-conformances, the samples must be re-analyzed.

10.7.3. **LLCCV**

10.7.3.1. Applicable to 6020A only

10.7.3.2. Same source as calibration

10.7.3.3. Frequency: Perform at a minimum at the end of the run

10.7.3.4. Criteria: ± 30%

10.7.3.5. Action upon failure:

10.7.3.5.1. If the LLCCV fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.3.5.2. If the LLCCV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.3.5.3. For all other non-conformances, the samples must be re-analyzed.

10.7.4. **CCB**

10.7.4.1. Frequency: A CCB is analyzed immediately following each CCV.

10.7.4.2. Criteria: The CCB result must fall within ± RL from zero.

10.7.4.3. Action upon failure:

10.7.4.3.1. If the CCB fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.4.3.2. If the CCB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.4.3.3. For all other non-conformances, the samples must be re-analyzed.

10.7.5. If a CCV and/or CCB has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then a second CCV and/or CCB may be analyzed without any adjustments to the instrument. If this CCV and/or CCB meet criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV and/or CCB does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e., initial calibration).

10.8. **Interference Check Standard (ICSA/ICSAB)**

10.8.1. Applicable to 6020 and 6020A only

10.8.2. The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions.

10.8.3. **ICSA**:

10.8.3.1. The ICSA contains only interfering elements. Refer to LIMS for the details of ICSA composition.
10.8.3.2. Custom multi-element ICS solutions must be used.
10.8.3.3. Elements known to be interferents on a required analyte must be included in the ICPMS run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICPMS runs.
10.8.3.4. Frequency: The ICSA must run with each initial calibration or every 12 hours whichever is shorter.
10.8.3.5. Criteria: ICSA results for the non-interfering elements with RL < 10 µg/L must fall within ± 2x RL from zero. ICSA results for the non-interfering elements with RL ≥ 10 µg/L must fall within ± 1xRL from zero.
10.8.3.6. Action upon failure:
10.8.3.6.1. For interfering elements:
10.8.3.6.1.1 If the ICSA fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
10.8.3.6.2. For non-interfering elements:
10.8.3.6.2.1 If the ICSA fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.8.3.6.2.2 If the ICSA fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.8.3.6.3. For all other non-conformances, the samples must be re-analyzed.

10.8.4. **ICSAB**:
10.8.4.1. The ICSAB contains analytes and interferents.
10.8.4.2. Refer to LIMS for the details of ICSAB composition.
10.8.4.3. Custom multi-element ICS solutions must be used.
10.8.4.4. Frequency: The ICSAB must run with each initial calibration or every 12 hours whichever is shorter.
10.8.4.5. Criteria: The ICSAB results must fall within 80% – 120% of the true value.
10.8.4.6. Action upon failure:
10.8.4.6.1. If the ICSAB fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
10.8.4.6.2. For all other non-conformances, the samples must be re-analyzed.

10.9. **Liner Dynamic Range (LDR)**
10.9.1. Prior to running the instrument, the upper limit of quantitation must be established for each analyte.
10.9.2. This upper limit is tested by running a standard containing high concentrations of the analytes against a calibration curve.
10.9.3. The concentration of the LDR standard is higher than the high calibration standard.
10.9.4. Frequency: Every 6 months
10.9.4.1. When requested by client, the LDR is run daily.
10.9.5. Criteria: ± 10%
10.9.5.1. If the LDR fails the criteria, the highest calibration standard is used as the upper limit for the

linear range.

10.10. **Calibration Sequence**

Tuning Standard
Initial Calibration (3 standards plus a blank)
ICV
ICB
LLICV (for 6020A only)
ICSA*
ICSAB*
CCV
CCB
LDR (Client Specific)
CCV
CCB
10 samples (analysis runs)
CCV
CCB
10 samples (repeat every 10 analysis runs)
LLCCV (Minimum at end of run. May be analyzed more frequently)
CCV
CCB
End

* If sequence time is longer than 12 hours, the ICSA and ICSAB standard must be re-analyzed.

11.0 PROCEDURE

11.1 The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
11.1.4 For 200.8 analyses, dissolved samples must be digested.

11.2 The soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.

11.3 Instrument conditions, including rinse times, must be the same for all standards and samples.

11.4 Internal standards are introduced to the standards and sample digestates by the instrument.

11.5 Load autosampler with standards and digestates in accordance with the sequence given in section 10

11.6 Analyze samples.

11.7 When analysis is completed, return unused digestate to proper storage area.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1. Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2. All measurements must fall within the defined linear range where spectral interference correction factors

are valid.

12.2.1. Dilute and reanalyze all samples for required analytes that exceed the linear range

12.2.1.1. For 200.8, any sample greater than 90% of the linear range must be diluted and re-analyzed.

12.2.2. Acid strength must be maintained in the dilution of samples.

12.3. The mass ions used for determination of the element of interest is given in Table 1 of this SOP

12.4. Tracer Calculations

12.4.1. Tracer Recovery: The LIMS calculates the tracer recovery using the following formula:

$$\frac{\text{Measured Tracer Concentration}}{\text{Actual Tracer Concentration}} = \text{Final Recovery}$$

12.4.1.1. Tracer Criteria: The tracer recovery must fall within 30-110%.

12.4.2. Final sample concentration (corrected for tracer recovery) is determined by the LIMS using the following formula:

$$\frac{\text{Measured Sample Concentration}}{\text{Tracer Recovery}} = \text{Final Sample Concentration}$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1. The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process in LIMS. The NCM process is described in SOP: ST-QA-0036.

13.2. Method Blank (MB)

13.2.1. Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.

13.2.2. Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted in the client notes.

13.2.3. Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.3.1. Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3. Laboratory Control Sample (LCS)

13.3.1. Acceptance Criteria: All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.

13.3.2. Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1. LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2. LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1. Analytes should be within control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.

13.4.2. Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1. MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration

13.4.2.1.1. If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.4.2.1.2. If the excursion is due to physically evident matrix interference, the data is reported with an NCM.

13.4.2.1.3. In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with an NCM.

13.4.2.1.4. When the MS/MSD recoveries and the %RPD are outside criteria, if the samples are non-homogenous, the data may be reported with an NCM. Otherwise, the batch is re-prepped and re-analyzed for the affected analytes.

13.5. Serial Dilution (SD)

13.5.1. Acceptance Criteria: The serial dilution results shall agree within ± 10% of the undiluted sample results, if the undiluted sample results are greater than 10 times the reporting limit. There is no criterion for sample results less than 10 times the reporting limit.

13.5.2. Corrective Action: Serial dilution failure is documented in an NCM and the reported data is flagged. If multiple analytes fail the serial dilution test, the analyst may re-prep and re-analyze the samples.

13.6. Post Digestion Spike (PDS)

13.6.1. Method 6020 and 6020A stipulates that a PDS be performed on the sample chosen for MS/MSD. A PDS is not required for 200.8.

13.6.2. **6020 Criteria**: The acceptance criteria is 75%-125%, UNLESS, other project/program criteria is given.

13.6.2.1. Corrective Action: Sample must be diluted and re-analyzed to compensate for matrix effect, until the PDS is within acceptable limits.

13.6.3. **6020A Criteria**: The acceptance criteria is 80%-120%, with a spike concentration between 10-100 times the MDL, UNLESS, other project/program criteria is given.

13.6.3.1. Corrective Action: There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with an NCM and noted in the report narrative.

13.6.4. The PDS is not reported in the data package unless a client project or program requires it. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.

13.7. Sample result evaluation

13.7.1. Dilutions

13.7.1.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.7.2. For samples requiring dilution an NCM is created to document the reason for the dilution.

13.7.3. Insufficient Sample

13.7.3.1. For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative.

13.8. Internal Recovery Standard (IS)

13.8.1. Criteria (for all samples and QC standards)

13.8.1.1. **6020:**

13.8.1.1.1. QC: 80-120%

13.8.1.1.2. Samples: 30-120%

13.8.1.2. **6020A**: 70%-140%

13.8.1.3. **200.8**: 60%-125

13.8.2. Action Upon Failure

13.8.2.1. Samples: If the criteria is not met, the sample should be diluted and re-analyzed until the

IS recoveries are within specified limits.

13.8.2.2. QC standards: If the criteria are not met, the analyst will review the data. If the sample internal standard recoveries are within control and the QC standard is within acceptable limits, it is apparent that whatever interference affected the internal standard for the QC standards has not affected the element bracketed by that internal standard based upon the criteria being met. If these specific occurrences are met then an NCM will be generated stating why the data is acceptable. Otherwise, samples linked to the QC standard will be re-analyzed

14.0 METHOD PERFORMANCE

14.1. Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.

14.2. Demonstration of Capability

14.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3. Training Qualification

14.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1. Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2. Waste Streams Produced by the Method

16.2.1. The following waste streams are produced when this method is carried out.

16.2.1.1. Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.1.2. Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid Rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 6020A

17.2. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma/Mass Spectrometry Method 200.8

17.3. PerkinElmer® ELAN 6100 Inductively Coupled Plasma Mass Spectrometer Hardware Guide
17.4. PerkinElmer® ELAN 6100 Software Kit
17.5. PerkinElmer® ELAN 9000 Hardware Guide
17.6. PerkinElmer® ELAN Version 3.0 Software Guide
17.7. Agilent 7500 Series MassHunter Workstation (G7200A) Operators Manual
17.8. Agilent 7500 Series ICP-MS Hardware Manual
17.9. Agilent 7700 Series ICP-MS Hardware Maintenance Manual
17.10. TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.11. TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.12. Associated SOPs, current revisions:
- 17.12.1. ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS
- 17.12.2. ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
- 17.12.3. ST-QA-0002, Standard and Reagent Preparation
- 17.12.4. ST-PM-0002, Sample Receipt and Chain of Custody
- 17.12.5. ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.12.6. ST-QA-0016, IDL/MDL Determination
- 17.12.7. ST-QA-0036, Non-conformance Memorandum (NCM) Process
- 17.12.8. ST-RC-0125, Determination of Technetium-99 Using Eichrom Resin

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1. Method 6020A stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed. Internal standards are used to monitor matrix interferences in all samples. Post spikes are done per specific QAPP or program requirements. Post-spikes using analytes other than the internal standards may be used if an analyst encounters a new or unusual matrix.

18.2. Method 6020A requires the analysis of a Lower Limit Quantitation Check Sample (LLQC) on an as needed basis, to establish and confirm the lowest quantitation limit. TestAmerica St. Louis fills this requirement with the quarterly running of a MDL verification standard which is taken through the entire sample preparation procedure.

19.0 CHANGES TO PREVIOUS REVISION

19.1. Updated formatting and spelling errors throughout SOP
19.2. Updated section 4.4 referring to the amount of an internal standard being used.
19.3. Added new instrument and gases used in section 6.0.
19.4. Added Lithium to section 7.0 as part of the new reagents and standards used.
19.5. Made reference to new instruments for calibration in section 10.0.
19.6. Add new list of tuning element for both instruments in section 10.5.
19.7. Updated the internal standard intensity throughout section 10.7 and section 10.8

19.8. Added new elements to table 2.
19.9. Rev. 18;
- 19.9.1. Added LLICV to definitions in section 3.2.
- 19.9.2. Removed Hydrogen Peroxide from Safety Section (included in prep SOP)
- 19.9.3. Added tuning solution to section 7.5.5.
- 19.9.4. Updated cone conditioning solution, make up and frequency of use.
- 19.9.5. Added clarification to tuning section 10.5.
- 19.9.6. Added Low Level initial calibration verification standards plus criteria to section 10.0.
- 19.9.7. Updated tables 1 and 2, added analytes, updated concentrations.
- 19.9.8. Added method 1311 MSA requirements information to section 9.7.
- 19.9.9. Spelling and grammatical corrections.

19.10. Rev. 19:
- 19.10.1. Updated Table III regarding QC Criteria limits.

19.11. Revision 20:
- 19.11.1. Updated section 1.3 adding reference to the Technetium-99 soil procedure.
- 19.11.2. Added formulas for determining the Tracer Recovery and the Final tracer Corrected Concentration to section 12.5.
- 19.11.3. Added instrument software and hardware to section 6.0.
- 19.11.4. Updated the PDS acceptance criteria in section 9.6.

19.12. Rev. 21:
- 19.12.1. Removed legacy text regarding MSA from Section 18 as MSA is not required by Method 6020A.

19.13. Rev. 22: (8/27/2013)
- 19.13.1. Updated section 1.3 to reflect the corrected Tc-99 SOP (ST-RC-0125)
- 19.13.2. Updated section 6. Hydrogen was removed
- 19.13.3. Updated section 9.7, replace QuantIMS wording with TALS wording
- 19.13.4. Updated section 10, used a more consistent format
- 19.13.5. Updated section 12.4. Removed references to a spreadsheet, added TALS
- 19.13.6. Updated section 13

19.14. Rev.23: (1/16/2015)
- 19.14.1. Made formatting and grammatical corrections
- 19.14.2. Corrected definitions in section 3 (removed IPC and CRI, added LRB)
- 19.14.3. Removed references to ASTM Method D5673-03
- 19.14.4. Added equipment and software for ICPMS 9000 to section 6
- 19.14.5. Removed the ICSA/ICSAB Table (was Table 2) and replaced references to it with instructions to look at LIMS (10.8.3.1 and 10.8.4.2)
- 19.14.6. Updated tuning criteria for Perkin Elmer ICPMS in sections 10.5.3.1.4 & 10.5.3.1.5
- 19.14.7. Updated section 10.8.3.5 , replace RL > 10ug/L with RL ≥10ug/L
- 19.14.8. Clarified criteria in section 10, 13 and 18
- 19.14.9. Replaced “client requirement sheet” with “client notes” in section 13.2.2
- 19.14.10. Removed reference to Marginal Exceedance in section 13.3
- 19.14.11. Corrected IS criteria in section 13.8.1
- 19.14.12. Updated instrument manuals in section 17
- 19.14.13. Added affected methods to section 18
- 19.14.14. Removed section 18.3, was same as section 18.4
- 19.14.15. Added reference to method 6020
- 19.14.16. Deleted references in section 9 to batch QC criteria, referenced section 13. Instrument QC criteria is in section 10.
- 19.14.17. updated formatting, to make the wording more consistent throughout the SOP and easier to read.
- 19.14.18. section 10: separated out the tuning for Perkin Elmer vs Agilent, for clarity.
- 19.14.19. Added reference to tc-99 prep via ST-RC-0125

19.15. Rev. 24: (06/22/2015)
- 19.15.1. Added Appendix 1 MSA instructions

Table 1
ANALYTICAL ISOTOPES

ELEMENT	7700 Tune Step	7500 Mass	7700 Tune Step	7700 Mass	6100 Mass	9000 Mass
Li	3	7	3	7	N/A	N/A
Be	3	9	3	9	N/A	N/A
B	3	11	3	11	N/A	N/A
Na	2	23	2	23	N/A	N/A
Mg	2	24	2	24	N/A	N/A
Al	2	27	2	27	N/A	N/A
Si	3	28	3	28	N/A	N/A
P	2	31	2	31	N/A	N/A
S	2	34	2	34	N/A	N/A
K	2	39	2	39	N/A	N/A
Ca	3	44	3	44	N/A	N/A
Ti	3	47	3	47	N/A	N/A
V	2	51	2	51	N/A	N/A
Cr	2	52	2	52	N/A	N/A
Mn	2	55	2	55	N/A	N/A
Fe	2	57	2	57	N/A	N/A
Co	2	59	2	59	N/A	N/A
Ni	2	60	2	60	N/A	N/A
Cu	2	63	2	63	N/A	N/A
Zn	2	66	2	66	N/A	N/A
As	2	75	2	75	N/A	N/A
Se	2	78	2	78	N/A	N/A
Sr	3	88	3	88	N/A	N/A
Y	2	89	2	89	N/A	N/A
Zr	2	90	2	90	N/A	N/A
Nb	2	93	2	93	N/A	N/A
Mo	3	95	3	95	N/A	N/A
Ru	2	101	2	101	N/A	N/A
Rh	2	103	2	103	N/A	N/A
Pd	2	105	2	105	N/A	N/A
Ag	3	107	3	107	N/A	N/A
Cd	3	111	3	111	N/A	N/A
Sn	3	118	3	118	N/A	N/A
Sb	3	121	3	121	N/A	N/A
Te	2	125	2	125	N/A	N/A
Cs	2	133	2	133	N/A	N/A
Ba	3	137	3	137	N/A	N/A
La	2	139	2	139	N/A	N/A
Ce	2	140	2	140	N/A	N/A
Pr	2	141	2	141	N/A	N/A
Nd	2	146	2	146	N/A	N/A
Sm	3	147	3	147	N/A	N/A
Hf	2	178	2	178	N/A	N/A
Ta	2	181	2	181	N/A	N/A
W	2	182	2	182	N/A	N/A
Re	3	185	3	185	N/A	N/A
Pt	2	195	2	195	N/A	N/A
Au	3	197	3	197	N/A	N/A
Tl	3	205	3	205	N/A	N/A
Pb	3	208	3	208	N/A	N/A
Bi	2	209	2	209	N/A	N/A
Th	3	232	3	232	N/A	N/A
Tc	3	99	N/A	N/A	99	99
U	N/A	N/A	3	236	236	236
U	N/A	N/A	3	235	235	235
U	N/A	N/A	3	234	234	234
U	N/A	N/A	3	233	233	233
U	3	238	3	238	238	238

Tune Step 2: Helium
Tune Step 3: No Gas (argon only)

Table 2
QC Criteria

Methods	6020	6020A	200.8
Corr Coeff.	>0.998	>0.998	
Tuning Res	<0.9amu	<0.9amu	≈ 0.75amu
Int Std	QC: 80-120% Samples: 30-120%	70-140%	60-125%
LCS	80-120%	80-120%	85-115%
ICV	90-110%	90-110%	90-110%
CCV	90-110%	90-110%	85-115%
PDS	75-125%	80-120%	N/A
MS	75-125%	75-125%	70-130%
LLICV	N/A	70-130%	N/A

Table 3

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS
BACKGROUND MOLECULAR IONS

Molecular Ion	Mass	Element Interferences*
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H_+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	
$^{40}ArH^+$	41	
CO_2^+	44	
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	
$ArOH^+$	57	
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se

* Method elements or internal standards affected by the molecular ions.

Table 4

MATRIX MOLECULAR IONS* No gas Mode Only

CHLORIDE		
Molecular Ion	Mass	Element Interference
$^{35}Cl0^+$	51	V
$^{35}Cl0H^+$	52	Cr
$^{37}Cl0^+$	53	Cr
$^{37}Cl0H^+$	54	Cr
$Ar^{35}Cl^+$	75	As
$Ar^{37}Cl^+$	77	Se

SULFATE		
Molecular Ion	Mass	Element Interference
$^{32}SO^+$	48	
$^{32}SOH^+$	49	
$^{34}SO^+$	50	V, Cr
$^{34}SOH^+$	51	V
SO_2^+, S_2^+	64	Zn
$Ar^{32}S^+$	72	
$Ar^{34}S^+$	74	

PHOSPHATE		
Molecular Ion	Mass	Element Interference
PO^+	47	
POH^+	48	
PO_2^+	63	Cu
ArP^+	71	

GROUP I, II METALS		
Molecular Ion	Mass	Element Interference
$ArNa^+$	63	Cu
ArK^+	79	
$ArCa^+$	80	

MATRIX OXIDES*		
Molecular Ion	Mass	Element Interference
TiO	62-66	Ni, Cu, Zn
ZrO	106-112	Ag, Cd
MoO	108-116	Cd

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

APPENDIX 1 – METHOD OF STANDARD ADDITION (MSA)

MSA is required if

1- Matrix spike recovery <50%, AND
2- Measured concentration is within 20% of the regulatory level

Regulatory Limits:

Arsenic	5mg/L
Barium	100mg/L
Cadmium	1 mg/L
Chromium	5 mg/L
Lead	5 mg/L
Mercury	0.2 mg/L
Selenium	1 mg/L
Silver	5 mg/L

How to run an MSA

1- Take 4 identical aliquots of test solution
2- Add increasing concentration of standard to 3 aliquots and add blank solution to 4th aliquot- all aliquots should be at same final volume
3- Perform analysis
4- Enter data into spreadsheet ORG-0034_TCLP_MSA
    a. Uses unweighted least-squares linear regression curve fit
    b. Calculates absolute value of x-intercept

Notes:

1- MSA spikes must come from the same analytical batch, analyzed on the same day.

The MSA curve must include the unspiked sample and 3 samples spiked with increasing concentrations of analytes

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS A ICSAB_00170

Reagent ID: MS A ICSAB_00170

		Expiration Date:	06/26/2015
Description:	Agilent ICSAB	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Buffington, Cory C
Storage Location:	Metals Standards Storage	Solvent:	2% HCL 2% HN03
Reagent Volume:	250.000 mL	Solvent Lot:	661194
Creation Date:	06/19/2015		
Open Date:			
Container(s):	663460		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Ag	MS CAL3 A_00006	02/11/2016	20.00000	ug/mL	10.00000	ug/L
As	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
B	MS CAL3 A_00006	02/11/2016	200.00000	ug/mL	100.00000	ug/L
Ba	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Be	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Cd	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Co	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Cr	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Cu	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Li	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Mn	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Ni	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Pb	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Se	MS CAL3 A_00006	02/11/2016	50.00000	ug/mL	25.00000	ug/L
Sm	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Sr	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Th	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Tl	MS CAL3 A_00006	02/11/2016	20.00000	ug/mL	10.00000	ug/L
U	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS A ICSAB_00170

Reagent ID: MS A ICSAB_00170

		Expiration Date:	06/26/2015
Description:	Agilent ICSAB	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Buffington, Cory C
Storage Location:	Metals Standards Storage	Solvent:	2% HCL 2% HN03
Reagent Volume:	250.000 mL	Solvent Lot:	661194
Creation Date:	06/19/2015		
Open Date:			
Container(s):	663460		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
V	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Zn	MS CAL3 A_00006	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Mo	MS CAL3 B_00007	02/11/2016	50.00000	ug/mL	2025.00000	ug/L
Sb	MS CAL3 B_00007	02/11/2016	50.00000	ug/mL	25.00000	ug/L
Sn	MS CAL3 B_00007	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Ti	MS CAL3 B_00007	02/11/2016	100.00000	ug/mL	2050.00000	ug/L
W	MS CAL3 B_00007	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Zr	MS CAL3 B_00007	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Au	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Bi	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Ce	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Cs	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
La	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Nd	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Pd	MS CAL3 ODD A_00005	02/11/2016	10.00000	ug/mL	5.00000	ug/L
Pr	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Pt	MS CAL3 ODD A_00005	02/11/2016	10.00000	ug/mL	5.00000	ug/L
Re	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Rh	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS A ICSAB_00170

Reagent ID: MS A ICSAB_00170

		Expiration Date:	06/26/2015
Description:	Agilent ICSAB	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Buffington, Cory C
Storage Location:	Metals Standards Storage	Solvent:	2% HCL 2% HN03
Reagent Volume:	250.000 mL	Solvent Lot:	661194
Creation Date:	06/19/2015		
Open Date:			
Container(s):	663460		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Ru	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Y	MS CAL3 ODD A_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Hf	MS CAL3 ODD B_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Nb	MS CAL3 ODD B_00005	02/11/2016	50.00000	ug/mL	25.00000	ug/L
Ta	MS CAL3 ODD B_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Te	MS CAL3 ODD B_00005	02/11/2016	100.00000	ug/mL	50.00000	ug/L
Al	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
Ca	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
Chlorine	MS ICSA_00017	03/31/2016	10000.00000	ug/mL	1000000.00000	ug/L
Fe	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
K	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
Mg	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
Mo	MS ICSA_00017	03/31/2016	20.00000	ug/mL	2025.00000	ug/L
Na	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
P	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
Sulfur	MS ICSA_00017	03/31/2016	1000.00000	ug/mL	100000.00000	ug/L
Ti	MS ICSA_00017	03/31/2016	20.00000	ug/mL	2050.00000	ug/L
Si	STD SI_00010	12/01/2015	1000.00000	ug/mL	500.00000	ug/L

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
MS CAL3 A_00006	MS CAL 3 A	ASTD	02/11/16	Inorganic Ventures	J2-MEB566024	TA-CAL-3	0.12500	mL
MS CAL3 B_00007	CAL 3 B	ASTD	02/11/16	Inorganic Ventures	J2-MEB566020	TA-CAL-3-2	0.12500	mL
MS CAL3 ODD A_00005	CAL 3 ODD	ASTD	02/11/16	Inorganic Ventures	J2-MEB566021	TA-CAL-3-ODD	0.12500	mL
MS CAL3 ODD B_00005	CAL 3 ODD B	ASTD	02/11/16	Inorganic Ventures	J2-MEB566022	TA-CAL-3-ODD-	0.12500	mL
MS ICSA_00017	ICSA	ASTD	03/31/16	Inorganic Ventures	J2-MEB533111	6020ICS-0A	25.00000	mL
STD SI_00010	SI 1000PPM	ASTD	12/01/15	INORGANIC VENTURE	G2-SI03029	CGSI1-1	0.12500	mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS INT STD_00006

Reagent ID: MS INT STD_00006

Type:	ASTD	Expiration Date:	11/01/2015
Description:	INT STD	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Souris, Matthew T
Storage Location:	Metals Standards Storage	Vendor:	Inorganic Ventures
Reagent Volume:	500.000 mL	Vendor Lot #:	H2-MEB547059
Creation Date:	08/19/2013	Vendor Cat #:	TA-INT-STD-REV-1
Open Date:	01/08/2015		
Container(s):	521050		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Au (IS)					100.00000	ug/mL
Ge Internal Standard					100.00000	ug/mL
Ho (IS)					100.00000	ug/mL
In Internal Standard					100.00000	ug/mL
Li-6 Internal Standard					100.00000	ug/mL
Sc (IS)					100.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS ICV 2_00004

Reagent ID: MS ICV 2_00004

Type:	ASTD	Expiration Date:	11/30/2015
Description:	ICV 2 - new	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Buffington, Cory C
Storage Location:	Metals Standards Storage	Vendor:	SPEX
Reagent Volume:	250.000 mL	Vendor Lot #:	27-169CR
Creation Date:	12/08/2014	Vendor Cat #:	ZITMO-51-250
Open Date:	12/08/2014		
Container(s):	520114		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Ag					40.00000	ug/mL
Ba					200.00000	ug/mL
Be					200.00000	ug/mL
Cd					200.00000	ug/mL
Co					200.00000	ug/mL
Cr					200.00000	ug/mL
Cu					200.00000	ug/mL
Li					200.00000	ug/mL
Mn					200.00000	ug/mL
Ni					200.00000	ug/mL
Pb					200.00000	ug/mL
Sm					200.00000	ug/mL
Sr					200.00000	ug/mL
Th					200.00000	ug/mL
Tl					40.00000	ug/mL
U					200.00000	ug/mL
V					200.00000	ug/mL
Zn					200.00000	ug/mL
Zr					200.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS ICV 1_00004

Reagent ID: MS ICV 1_00004

Type:	ASTD	Expiration Date:	11/30/2015
Description:	ICV 1 - new	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Buffington, Cory C
Storage Location:	Metals Standards Storage	Vendor:	SPEX
Reagent Volume:	250.000 mL	Vendor Lot #:	27-168CR
Creation Date:	12/08/2014	Vendor Cat #:	ZITMO-50-250
Open Date:	12/08/2014		
Container(s):	520113		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
As					200.00000	ug/mL
B					400.00000	ug/mL
Mo					100.00000	ug/mL
Sb					100.00000	ug/mL
Se					100.00000	ug/mL
Sn					200.00000	ug/mL
Ti					200.00000	ug/mL
W					200.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS CAL3 A_00006

Reagent ID: MS CAL3 A_00006

Type:	ASTD	Expiration Date:	02/11/2016
Description:	MS CAL 3 A	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Buffington, Cory C
Storage Location:	Metals Standards Storage	Vendor:	Inorganic Ventures
Reagent Volume:	500.000 mL	Vendor Lot #:	J2-MEB566024
Creation Date:	02/11/2015	Vendor Cat #:	TA-CAL-3
Open Date:	02/11/2015		
Container(s):	559278		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Ag					20.00000	ug/mL
As					100.00000	ug/mL
B					200.00000	ug/mL
Ba					100.00000	ug/mL
Be					100.00000	ug/mL
Cd					100.00000	ug/mL
Co					100.00000	ug/mL
Cr					100.00000	ug/mL
Cu					100.00000	ug/mL
Li					100.00000	ug/mL
Mn					100.00000	ug/mL
Ni					100.00000	ug/mL
Pb					100.00000	ug/mL
Se					50.00000	ug/mL
Sm					100.00000	ug/mL
Sr					100.00000	ug/mL
Th					100.00000	ug/mL
Tl					20.00000	ug/mL
U					100.00000	ug/mL
V					100.00000	ug/mL
Zn					100.00000	ug/mL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MS CAL 1 A _ 00001

Reagent ID: MS CAL 1 A_00001

Type:	ASTD	Expiration Date:	02/23/2016
Description:	MS CAL 1 A	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Buffington, Cory C
Storage Location:	Metals Standards Storage	Vendor:	Inorganic Ventures
Reagent Volume:	500.000 mL	Vendor Lot #:	J2-MEB566091
Creation Date:	02/23/2015	Vendor Cat #:	TA-CAL-1
Open Date:	02/23/2015		
Container(s):	567607		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Ag					2.00000	ug/mL
As					10.00000	ug/mL
B					50.00000	ug/mL
Ba					2.00000	ug/mL
Be					0.50000	ug/mL
Cd					0.50000	ug/mL
Co					2.00000	ug/mL
Cr					10.00000	ug/mL
Cu					1.00000	ug/mL
Li					5.00000	ug/mL
Mn					2.00000	ug/mL
Ni					5.00000	ug/mL
Pb					3.00000	ug/mL
Se					5.00000	ug/mL
Sm					10.00000	ug/mL
Sr					5.00000	ug/mL
Th					2.00000	ug/mL
Tl					2.00000	ug/mL
U					1.00000	ug/mL
V					10.00000	ug/mL
Zn					10.00000	ug/mL

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Title: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION [SW846 7470A; MCAWW 245.1]

Approvals (Signature/Date):

Kristen Ely — 8/21/14 Kristen Ely — Date Inorganics Department Manager	Michael Ridenhower — 8/21/14 Michael Ridenhower — Date Health & Safety Manager / Coordinator
Marti Ward — 8.21.14 Marti Ward — Date Quality Assurance Manager	Elaine Wild — 8/21/14 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-MT-0005 Rev. 14

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1.0 SCOPE AND APPLICATION

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1.

1.2 CVAA analysis provides for the determination of total mercury (organic and inorganic).

1.3 Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, TCLP, EP and other leachates/extracts. All matrices require sample preparation prior to analysis.

1.4 Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.3 Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.4 Total Metals: The concentration determined on an unfiltered sample following digestion.

3.5 EPA and SW methodology use different terminology. This SOP references the SW 846 terminology:

- 3.5.1 The ICV satisfies the QCS requirements found in method 245.1.
- 3.5.2 The LCS satisfies the requirements of the LFB found in method 245.1.
- 3.5.3 The CCV satisfies the requirements of the IPC found in method 245.1.

4.0 INTERFERENCES

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Seawaters, brines and industrial effluents which are high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.5 Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3 Primary Materials Used

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limits (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Contact with other material may cause fire. Liquid and mist cause severe burns to all body tissue.
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 Mg/M3-(TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored

Material (1)	Hazards	Exposure Limits (2)	Signs and Symptoms of Exposure
	Poison		breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness. Sulfuric acid is extremely corrosive. Liquid and mist cause severe burn to all body tissue.
Hydrochloric Acid	Poison Corrosive	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Hydrochloric acid is corrosive. Liquid and mist causes severe burn to all body tissue.
Potassium permanganate	Oxidizer	5 mg/m3 (Ceiling)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent. Contact with other materials may cause fire. Causes burns to any area of contact. Harmful if swallowed or inhaled.
Hydroxylamine Hydrochloride	Poison Corrosive	None established	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache nausea and vomiting. Skin contact may cause irritation and burns. Eye contact may cause severe irritation and corneal damage. Causes burns to any area of contact. Harmful if swallowed of inhaled.
Mercury	Poison Corrosive	0.1 mg/m3 (TWA)	Inhalation symptoms include sore throat, coughing, pain, tightness in chest, breathing difficulties, shortness of breath, headache, muscle weakness, anorexia, gastrointestinal disturbance, ringing in the ear, liver changes, fever, bronchitis and pneumonitis. Skin contact produces redness and pain. May be absorbed through skin with symptoms paralleling those of inhalation. Eye contact symptoms include redness, pain, and blurred vision. Causes burns to skin, eyes, and respiratory tract. May be fatal if swallowed or inhaled. Harmful if absorbed through skin.
Potassium Persulfate	Oxidizer	0.1 mg/m3 (TWA)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis. Contact with other material may cause fire. Harmful if swallowed.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.
STEL – Short Term Exposure Limit
TWA – Time Weighted Average
Ceiling – At no time should this limit be exceeded.

6.0 EQUIPMENT AND SUPPLIES

6.1 Temperature controlled hot block (capable of maintaining a temperature of 95 ± 3°C).
6.2 Leeman Labs Mercury Analyzer
6.3 50 ml plastic containers, or equivalent
6.4 Argon gas supply, welding grade or equivalent.
6.5 Calibrated automatic pipettes or Class A glass volumetric pipettes.
6.6 Class A volumetric flasks.
6.7 Thermometer (capable of accurate readings at 95 ± 3°C).
6.8 Disposable cups or tubes.
6.9 Hydra II AA software: Envoy Version 2.8
6.10 Hydra AA Software: WinHg Version 1.4

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3 Nitric acid (HNO_3), concentrated, analytical reagent grade (AR) or better.

7.4 Hydrochloric acid concentrated, analytical reagent grade (AR) or better

7.5 Sulfuric acid (H_2SO_4), concentrated, analytical reagent grade (AR) or better

7.6 Stannous chloride: Dissolve 100g of stannous chloride into 1000 mL of 10% HCl.

7.7 Sodium chloride-hydroxylamine sulfate solution: Add 12 g of sodium chloride and 12 g of hydroxylamine sulfate to every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.

7.8 Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.

7.9 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.

7.10 Stock (100 ppm) mercury standards (in 5% HNO_3) are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.11 Intermediate mercury standard (0.1 ppm): Take 0.1 mL of the stock mercury standard and dilute to 100 mL with reagent water. The intermediate standard must be made daily and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.12 The calibration standards must be prepared fresh daily from the intermediate standard by transferring 0, 0.06, 0.15, 0.3, 1.5 and 3.0 mL aliquots of the intermediate mercury standard into 30 mL flasks and diluting to volume with reagent water.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.13 The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.14 All standards must be processed through the entire analytical procedure including sample preparation (for 245.1)

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Sample collection is not performed by the laboratory for this analysis.

8.2 Sample shipments received by the laboratory will be reviewed against the Sample Acceptance Policy described in SOP ST-PM-0002, "Sample Receipt and Chain of Custody."

8.3 The sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.4 Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

9.1.2 Conditions must be the same for all standards, samples and QC samples.

9.1.3 Each analytical batch may contain up to 20 environmental samples, a <u>method blank</u>, and a single <u>Laboratory Control Sample</u> (LCS), and Matrix Spike/Matrix Spike Duplicate (MS/MSD). In the event that there is insufficient sample to analyze a duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 **Method Blank**

9.2.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.2.2 A method blank must be included with each batch of samples. The matrix for aqueous is reagent (DI) water.

9.2.3 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).

9.3 **Laboratory Control Sample**

9.3.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every batch.
9.3.3 The water LCS is reagent (DI) water fortified with mercury.

9.4 **Matrix Spike/Matrix Spike Duplicate**
9.4.1 Definition: Two aliquots of a field sample to which a known amount of target analyte(s) is added.
9.4.2 Additional MS/MSD's do not count towards the 20 samples in an analytical batch.
9.4.3 Matrix spikes are performed every 10 samples for EPA 245.1.

9.5 **Sample Duplicates**
9.5.1 Definition: Sample Duplicate – a separate aliquot of a field sample taken through the entire analytical process.
9.5.2 Additional Sample Duplicates do not count towards the 20 samples in an analytical batch.
9.5.3 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed.

9.6 **Serial Dilution**
9.6.1 Definition: a sample is diluted 1:5 and reanalyzed to assess the present of a matrix interference.
9.6.2 A serial dilution is performed with every analytical batch.
9.6.3 Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences.

9.7 **Post Digestion Spikes**
9.7.1 Definition: A known amount of mercury is added to the sample chosen for serial dilution to bring the concentration of mercury to 2 to 5 times the original concentration. If the sample's mercury concentration is below the detection limit, spike at 20 times the detection limit.
9.7.2 At client's request, a post digestion spike is performed when the serial dilution test fails.
9.7.2.1 Post digestion spike recovery criteria: 85%-115%.

9.8 **Method of Standard Addition (MSA)**
9.8.1 Definition: This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.
9.8.1.1 Client Requirement Checklists - Certain clients may require specific project or program QC which may supersede the SOP requirements. These are documented on the client Requirement Checklist or Client Requirement Memos.
9.8.2 MSA's are not considered normal batch QC and if required by the client, must appear on the Client Requirement Checklist.

9.9 **Procedural Variations/ Nonconformance and Corrective Action**
9.9.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
9.9.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration
10.1.1 Calibration standards must be processed through the preparation procedure.
10.1.2 Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.1.3 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.1.4 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration.

10.1.5 Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the reporting limit. Analyze standards in ascending order beginning with the blank.

10.1.6 The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.1.7 If the calibration curve does not meet method requirements, another calibration curve should be performed.

10.2 Initial Calibration Verification (ICV)

10.2.1 An ICV is a second source verification of the initial calibration.

10.2.2 An ICV must be performed with every initial calibration.

10.2.3 If the ICV fails to meet criteria ±5% for Method 245.1 or ±10% for method 7470A, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

10.2.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.3 Initial Calibration Blank (ICB)

10.3.1 Analyze the initial continuing calibration blank (ICB) immediately following the ICV.

10.3.2 The absolute value of the result must be less than or equal to the reporting limit (RL). If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument if necessary.

10.4 Low Level Check (LLC) aka CRA (When requested by client)

10.4.1 Can be the same source as your calibration.

10.4.2 Must be performed with every initial calibration.

10.4.3 Must fall within 30% of the true value.

10.4.4 Must be spiked at RL

10.4.5 If the LLC does not meet QC criteria, the analysis must be terminated, the problem corrected and the instrument recalibrated

10.5 Continuing Calibration Verification (CCV)

10.5.1 A CCV is analyzed after every 10 samples and at the end of the analytical sequence run.

10.5.2 The CCV for EPA 7470A must fall within 20% of the true value for that solution; for EPA 245.1, the CCV must fall within 10% of the true value.

10.5.3 If the CCV does not meet QC criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

10.6 Continuing Calibration Blank (CCB)

10.6.1 Analyze a continuing calibration blank (CCB) immediately following the CCV.

10.6.2 The absolute value of the result must be less than or equal to the reporting limit (RL). If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument if necessary.

11.0 PROCEDURE

11.1 All calibration and calibration verification standards (ICV, ICB, LLC, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.2 Calibration Standard/Verification Standard Preparation:

11.2.1 Transfer 0, 0.06, 0.15, 0.3, 1.5, 3.0 mL aliquots of the intermediate standard onto a series of sample digestion bottles, and bring to 30 mL final volume with DI water.

11.2.2 Transfer 0.75 of QC intermediate for ICV, and bring to 30 mL final volume with DI water.

11.2.3 Transfer 1.5 ml of QC intermediate for CCV, and bring to 30 mL final volume with DI water.

11.2.4 Transfer 0.06ml of calibration intermediate for LLC and bring to 30ml to final volume with DI.

11.3 Sample and Batch QC Preparation:

11.3.1 Transfer 30 mL aqueous sample, 6mls for TCLP leachate, or 1 ml for CWET leachate to a pre-labled sample digestion tube. If not already at 30 mL, bring up to a final volume of 30 mls with DI water.

11.3.1.1 Spike the aqueous MS/MSD aliquots with 0.3 mL of the intermediate mercury standard.

11.3.1.2 Spike the TCLP or CWET MS/MSD aliquot with 1.5 mL of the intermediate mercury standard.

11.3.2 For the aqueous method blank, transfer 30 mL DI water to a pre-labeled sample digestion tube. For TCLP and CWET method blanks, use 30 ml of the appropriate leaching fluid.

11.3.2.1 Use 6mls of TCLP fluid diluted to 30mls with DI water.

11.3.2.2 Use 1ml of c-wet fluid diluted to 30mls with DI water.

11.3.3 For the aqueous LCS, spike 0.3 mL of the QC intermediate mercury standard into a pre-labeled digestion tube and bring to a 30 mL final volume with DI water. For TCLP and CWET LCS, spike 1.5 mL of QC the intermediate mercury standard into a pre-labeled digestion tube and bring to a 30 mL final volume with the appropriate leaching fluid.

11.4 Digestion:

11.4.1 Add 1.5 ml (5ml if 100 ml final volume) of concentrated H_2SO_4 and .75 ml (2.5 ml if 100 ml final volume) of concentrated HNO_3 mixing after each addition.

11.4.2 Add 4.5 ml (15 ml if 100 ml final volume) of potassium permanganate solution.

11.4.2.1 Permanganate color must persist for at least 15 minutes. Additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If additional portions of permanganate solution are needed, an equivalent portion must also be added to all QC and client samples being prepped to match the highest amount added to any one sample. If after the addition of up to 25 mL additional permanganate the color does not persist, sample dilution prior to reanalysis may be required.

11.4.2.2 When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all other associated samples and standards in the run. *In instances, where this is not feasible, the addition of additional reagent can be achieved without affecting the sample volume through the addition of small portions, 0.5g aliquots, of solid potassium permanganate.*

11.4.3 Add 2.4 mL (8 ml if 100 ml final volume) of potassium persulfate solution and heat for two hours in a hot block at 95 $\pm$ 3°C.

11.4.4 Cool samples.

11.4.5 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.5 Sample Analysis:

11.5.1 Add 2 mL of sodium chloride-hydroxylamine sulfate solution (6ml if 100ml final volume is utilized) to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains).

11.5.2 Automated determination: Follow instructions provided by instrument manufacturer.

11.5.3 Check Stannous chloride solution volume is sufficient to cover sample run.

11.5.3.1 Stannous chloride is introduced to the sample by the instrument.

11.5.4 The following analytical sequence must be used with 7470A and 245.1:

Instrument Calibration (5pt plus blank)
ICV
ICB
LLC
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

11.5.5 If the instrument analysis is stopped for any reason, the run may be resumed. The resumed run must begin with CCV/CCB only if the run has been stopped for more than 2 hours.

11.5.6 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; re-sloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis LQM.

12.2 Appropriate factors must be applied to sample values if dilutions are performed.

12.3 Sample results should be reported with up to three significant figures in accordance with the significant figure policy.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (MB)

13.2.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.

13.2.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements memo or in the comment section of LIMS.

13.2.3 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.3.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.4 Acceptance Criteria: All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.5 Corrective Action for LCS not meeting acceptance criteria:

13.5.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.5.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.6.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.6.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.6.2.1 MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration

13.6.2.1.1 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.6.2.1.2 If the excursion is due to a physically evident matrix interference, the data is reported with an NCM.

13.6.2.1.3 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with and NCM.

13.6.2.1.4 When the MS/MSD recoveries and the %RPD are outside criteria, if the sample is non-homogenous, data may be reported with an NCM. Otherwise, the batch is re-prepped and re-analyzed for the affected analytes.

13.7 Serial Dilution (SD)

13.7.1 The serial dilution results shall agree within ± 10% of the undiluted sample results, if the undiluted sample results are greater than 10 times the reporting limit. There is no criteria for sample results less than 10 times the reporting limit.

13.7.2 Corrective Action: Serial dilution failure is documented in an NCM and the reported data is flagged.

13.8 Post Digestion Spike (PDS)

13.8.1 The method stipulates that a PDS be performed on the sample chosen for MS/MSD.

13.8.2 There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with a Non-conformance memo (NCM) and noted in the report narrative.

13.8.3 The PDS is not reported in the data package unless a client project or program requires it. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.

13.9 Sample result evaluation

13.9.1 Dilutions

13.9.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.9.1.2 For samples requiring dilution an NCM is created to document the reason for the dilution.

13.9.2 Insufficient Sample

13.9.2.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

14.1 Method performance data, reporting limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of capability

14.2.1 Initial and continuing Demonstrations of Capability requirements are established in the ST-QAM.

14.3 Training qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial Demonstration of Capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" .

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury). (Source method.)

17.2 "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S. EPA Method 245.1, Revision 3.0 (1994). (Source method.)

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision

17.4 Corporate Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs, current revisions

17.5.1 ST-IP-0004, Labware Preparation for Inorganic and Trace Metals Analysis
17.5.2 ST-PM-0002, Sample Receipt and Chain of Custody
17.5.3 ST-QA-0002, Standard and Reagent Preparation
17.5.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.5.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.5.6 ST-QA-0016, IDL/MDL Determination
17.5.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 Modifications/Interpretations from reference method.
18.2 Modifications from both 7470A and 245.1.
18.2.1 The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
18.2.2 This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

18.3 Modifications from Method 7470A
18.3.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

18.4 Modifications from 245.1
18.4.1 Method 245.1 Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. TestAmerica reports all Hg results under this SOP to two significant figures.

18.5 Method SW846 1311 requires that the Method Standard Addition (MSA) be used when matrix spike recovery is less than 50% and the measured sample results is within the range of 80-120% of the Toxicity Characteristics Limit.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated section 10.0 by adding Low Level Check instructions.
19.2 Added section 11.2.4, pre of Low Level Calibration Check.
19.3 Added Low Level Checks to the Instrument Calibration list in section 11.5.4.
19.4 Rev. 9;
19.4.1 Updated post digestion spike section 9.7; removed MSA requirements
19.4.2 Removed section 18.3.2: redundant ICV criteria.
19.4.3 Added Method 1311 MSA requirements, section 18.5.
19.5 Rev. 10;
19.5.1 Updated the spike recovery acceptance criteria for methods EPA 245.1 and 7470A in section 13.4.
19.6 Rev. 11:
19.6.1 Updated instrument software used in section 6.0.
19.6.2 Updated permanganate solution needed for method blank and LCS in section 11.4.

19.7 Rev. 12:

19.7.1 Updated requirement for prepping all client and QC samples in section 11.4.2.1

19.8 Rev. 13:

19.8.1 Updated hot block temperate in section 6.0 and 11.4.

19.9 Rev. 14:

19.9.1 Removed references to QuantIMS and Clouseau through out.

19.9.2 Updated section 13 with corrective actions taken

19.9.3 Updated section 15

19.9.4 Removed trouble shooting and added to ST-QA-0024

19.10 Rev 15:

19.10.1 Updated 13.2.2

19.10.2 Added potential NCM for MS/MSD failure in section 13.6

19.10.3 Minor grammatical changes

19.10.4 Section 13.8 (PDS) removed “if PDS fails, a SD is performed” A SD is done with every batch.

Title: DETERMINATION OF GROSS ALPHA/BETA ACTIVITY

Approvals (Signature/Date):

Sarah C. Bernsen 2·12·14	Michael Ridenhower 2/12/14
Sarah Bernsen — Date	Michael Ridenhower — Date
Radiochemistry Prep Supervisor	Health & Safety Manager / Coordinator
Marti Ward 2·11·14	Elaine Wild 2/13/14
Marti Ward — Date	Elaine Wild — Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0020 Rev. 17

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1.0 SCOPE AND APPLICATION

1.1 This procedure applies to the preparation and analysis of samples for gross alpha and/or beta radioactivity in air filters, water, soil/sediment, oil and vegetation samples.

1.2 This SOP is based on EPA Method 900.0, SW-846 Method 9310 and DOE RP-710.

1.3 For water samples containing high concentrations of dissolved solids (> 500 ppm), see SOP ST-RC-0021 for analysis of gross alpha radioactivity.

1.4 The reporting limits, method detectable activities and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 An aliquot of aqueous sample is evaporated to dryness in a glass beaker after the addition of concentrated nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tared counting planchet.

2.2 For the activity of dissolved matter, an aliquot of aqueous sample is filtered through a 0.45-µm membrane filter. The filtrate is evaporated to dryness in a glass beaker after the addition of concentrated nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tarred counting planchet.

2.3 For the activity of suspended matter, an aliquot of aqueous sample is filtered through a 0.45-µm membrane filter. The filter is transferred to a counting planchet.

2.4 Air filter samples are counted for gross alpha and/or beta activity without further processing if the filter is less than 2 inches diameter. If the filter is greater than 2-inch diameter, the sample is digested per ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota , Oil and Grease Samples for Actinide Analysis" and then an aliquot prepared like a liquid.

2.5 Solid samples can be analyzed for gross alpha and/or beta activity as a dry powder. If Method RP710 (for total dissolution) is required, an acid leach is performed per ST-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis". The digestate is then treated like a liquid**.**
NOTE: Total Sample Dissolution can also be done using Hydrofluoric acid, Hydrochloric acid and Nitric acid as in section 11.8.

2.6 Oil samples are ashed in a muffle furnace, then dissolved in nitric acid and transferred to a glass beaker where they are converted to nitrate salts using concentrated nitric acid. The sample is then transferred to a planchet using 4 M nitric acid.

2.7 The sample residue is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 In this method for gross alpha and gross beta measurement, the radioactivity of the sample is not separated from the solids of the sample. The solid concentration may adversely affect sensitivity of the method.

4.2 For a 2-inch diameter counting planchet (20 cm^2), an aliquot containing 100 mg of dissolved solids would be the maximum aliquot size for that sample which should be evaporated and counted for gross alpha or gross beta activity.

4.3 Radionuclides that are volatile under the sample preparation conditions of this method can not be measured. Other radionuclides may also be lost during the sample evaporation and drying (such as tritium and some chemical forms of radioiodine). Some radionuclides, such as the cesium and technetium radioisotopes, may be lost when samples are heated to dull red color. Such losses are limitations of the test method.

4.4 Moisture absorbed by the sample residue increases self absorption and, if uncorrected, leads to low-biased results. Hygroscopic sample matrices may not remain at a constant weight after being dried and exposed to the atmosphere before and during counting. Those types of water samples need to be heated to a dull red color for a few minutes to convert the salts to oxides.

4.5 Heterogeneity of the sample residue in the counting planchet interferes with the accuracy and precision of the method.

4.6 Gross Alpha and Gross Beta activity does not identify the radionuclide that is present. Instead, the activity is referenced as equivalent to Th-230 for Gross Alpha and Sr-90/Y-90 for Gross Beta.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-(TWA) 4 ppm-(STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-(Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid	Poison Corrosive Dehydrator	3 ppm-(TWA)	Severely corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical Balance (4 - or 5 - place).

6.2 Beakers: Glass and Teflon, various sizes. Please consult SOP: ST-RC-5006 “Decontamination of Laboratory Glassware, Labware, and Equiptment.”

6.3 Counting planchets, stainless steel, flat and ridged, 5.0 cm (2.0"), cleaned per ST-RC-0002, “Preparation of Stainless Steel Planchets for Radiochemistry Analyses.”

6.4 Desiccator with desiccant, Dri-Rite or equivalent.

6.5 Drying oven with thermostat set at 105° C ± 5 °C.

6.6 Filter paper: ashless, Whatman #41 or ashless paper pulp, and 0.45-µm membrane.

6.7 Hot plate

6.8 Pipettes

6.9 Muffle oven

6.10 Mod block

6.11 Tongs or forceps

6.12 Double sided tape or Self-adhesive dots

6.13 Spatula

6.14 Aluminum weighing pans

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Reagents are prepared from reagent grade chemicals, unless otherwise specified below, and reagent water.

7.3 Deionized Water, obtained from the Milli-Q unit.

7.4 Nitric acid, concentrated (16N HNO_3)

7.4.1 4 N Nitric acid (4N HNO_3) - Add 250 ml of 16 N HNO_3 to 750 ml of reagent water and mix well.

7.5 Hydrofluoric acid, concentrated (29 N HF)

7.6 Hydrochloric acid, concentrated (12 N HCl)

7.7 Salt Solution: $NaHCO_3$ – 22 g, KCL – 0.80 g, $MgCl_2 \cdot 6H_2O$ – 22 g, Na_2SO_4 – 34.2 g add to 500 mL of DI water. Stir on stir plate until dissolved. Bring final volume up to 1 L with DI.

7.8 Salt, NaCl, granular.

7.9 Thorium-230 for LCS and matrix spikes, calibrated - NIST traceable, diluted to approximately 20 dpm/mL.

7.10 Strontium-90 for LCS and matrix spikes, calibrated - NIST traceable, in equilibrium with Yttrium 90, diluted to approximately 20 dpm/ml.

7.11 Sodium Bicarbonate, $NaHCO_3$ powder.

7.12 Potassium Chloride, KCl

7.13 Sodium Sulfate, $NaSO_4$ crystals

7.14 Magnesium Chloride Hexahydrate, $MgCl_2 \cdot 6H_2O$, crystals

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

8.3.1 The pH of aqueous samples is checked upon receipt by the Sample Control Department. The pH does not require re-checking prior to analysis.

8.3.2 Aqueous samples acidified upon receipt (designated by label on the bottle) do require a check of the pH prior to analysis.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a <u>method blank (MB)</u>, a <u>Laboratory Control Sample</u> (LCS), and <u>Sample Duplicate</u>. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water. Prepare a method blank of DI water equivalent to the target volume of 200 mL.

9.2.4 For Soil analyses, the method blank is comprised of salt.

9.2.5 For Oil analyses, the method blank is comprised of shredded filter paper in a crucilble.

9.2.6 For non-digested filters, a prepared method blank is provided by the count room.

9.2.7 For leached analyses, the method blank is comprised of the leaching acid.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Strontium 90 for beta and Thorium 230 for alpha. Add 0.7 mL of salt solution for mass.

9.3.4 For Soil analyses, the LCS is comprised of a known solid reference material :National Bureau of Standards, SRM 4353, Rocky Flats Soil #1.

9.3.5 For Oil analyses, the LCS is comprised of shredded filter paper fortified with Strontium 90 for beta and Thorium 230 for alpha.

9.3.6 For non-digested filters, the LCS is provided by the count room.

9.4 **Matrix Spike (MS)/Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate (SD)**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of a LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance calibration must be checked daily when used. Refer to SOP ST-QA-0005, “Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes Procedure.

10.2 For analytical instrumentation calibration, see SOP: ST-RD-0403, “Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System”.

11.0 PROCEDURE

11.1 If the activity of dissolved matter in an aliquot of aqueous sample is to be determined.

11.1.1 Filter the desired aliquot through a 0.45-µm membrane filter and proceed with aqueous sample preparation.

11.2 If the activity of suspended matter of an aliquot of aqueous sample is to be determined.

11.2.1 Filter the desired aliquot through a 0.45-µm membrane filter, and proceed with filter sample preparation.

11.3 Aqueous Sample - Total Solid Screen

11.3.1 Record sample preparation data in Gross Alpha/Beta (GAB) Solid Screen Excel program (RAD-0052). Weigh the empty beaker and record weight (under the tare weight header)

11.3.2 Shake the sample container thoroughly.

11.3.2.1 If alpha and beta are to be determined simultaneously from a single aliquot, the lowest net residue weight limit applies.

11.3.3 Measure a 20 mL aliquot into a pre-weighed beaker.

11.3.4 Add 10 mL of concentrated Nitric acid.

11.3.5 Evaporate to dryness using a hot plate, do not allow the sample to splatter.

11.3.6 Remove from heat and allow to cool to room temperature.

11.3.7 Add 10 mL concentrated Nitric acid.

11.3.8 Evaporate to dryness using a hot plate. Do not allow the sample to splatter.

11.3.9 Remove from heat and allow to cool in desicator for a minimum of 30 minutes.

11.3.10 Reweigh the beaker in GAB Solid Screen Excel (RAD-0052) program and record weight (under the gross weight header)

11.3.10.1 By estimating the solid content of the sample, the program will provide the target aliquot .

11.3.10.2 If GAB Solid Screen Excel program is not available use the formula found in 12.2.

11.3.10.3 From the net residue weight and sample volume used, determine the sample volume required to meet the target residue weight using the formula given in step 12.2, with a target weight of 80 mg alpha/beta dried residue on the planchet **(sample weights should not exceed 100 mg, if sample weights exceed 100 mg an aliquot of the dried residue should be taken after redissolving in 4 N nitric acid. Dilutions are noted on the worksheet. If it is not practical to redissolve the residue the sample should be redone using less volume. If it is not practical to redissolve or restart the sample, check with the count room supervisor or designee to verify that the sample weight fits on the current alpha curve before counting.)**. If only Gross Beta is being performed, the target weight is to 160 mg. Compare the calculated volume to meet the weight limitation with the volume required to ensure that the MDA is below the Reporting Limit. The volume for analysis is the smaller of the two volumes.

11.4 Aqueous Sample Gross Alpha/Beta

11.4.1 Initiate sample preparation worksheet.

11.4.2 Shake the sample container thoroughly.

11.4.3 Measure a volume of sample, previously determined in section 11.3, into an appropriately sized beaker. Record volume of sample used.

11.4.3.1 If it is determined (in step 11.3) that only a small volume of sample is required, additional volume may be added in small aliquots directly to the beaker used to determine the volume needed to achieve the target sample weight.

11.4.4 Prepare a method blank, LCS and MS.

11.4.5 Add 10 mL of concentrated Nitric acid to all samples and QC.

11.4.6 Evaporate to near dryness using a hot plate. Do not allow the sample to splatter.

11.4.7 Remove from heat and allow to cool to room temperature.

11.4.8 Add 10 mL concentrated Nitric acid.

11.4.9 Evaporate to near dryness using a hot plate. Do not allow the sample to splatter.

NOTE: Some samples with difficult matrices may require steps 11.4.7 through 11.4.9 to be repeated until the sample residue does not change in appearance.

11.4.10 Remove from heat and allow to cool to room temperature.

11.4.11 Add 10 mL of 4 N nitric acid to wash down the sides of the beaker.

11.4.12 Heat on hot plate to dissolve sample residue and reduce volume to approximately 5-7 mL.

11.4.13 Transfer the sample to a ridged stainless steel planchet.

11.4.14 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet.

11.4.15 Evaporate planchets to dryness on a hot plate. Do not allow the sample to splatter.

11.4.16 Remove sample planchets from hot plate.

11.4.17 Dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours, if sample appears hygroscopic. If not hygroscopic proceed to step 11.4.18.

11.4.18 Cool planchets in a desiccator for a minimum of 30 minutes.

11.4.19 Weigh the cooled planchets and record final weight(s).

11.4.19.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.4.19.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.4.19.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).

11.4.20 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.5 Oil Sample

11.5.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.

11.5.2 Fill a 50 mL beaker ¼ full with confetti made from Whatman No. 41 filter paper or ashless paper pulp.

11.5.3 Place beaker on analytical balance, then record the weight in appropriate sample worksheet.

11.5.4 Weigh to the nearest 0.0001 g, approximately 0.1-1gram of the oil sample onto the shredded filter paper. Record the sample weight.

11.5.5 Cover with a crucible lid.

11.5.6 If the sample is a mixture of oil and water or is a sample spiked with an aqueous solution, evaporate the water on a hot plate before muffling. Do not allow residue to "bake" on hot plate. A programmable muffle program may also be used to dry the water before ramping the temperature.

11.5.7 Ramp oven to approximately 600° C and hold there for four hours.

11.5.8 Turn off the muffle oven, crack open the door, and allow the sample to cool to room temperature.

11.5.9 Add approximately 7 mL of 4 N HNO_3 to the residue in the beaker.

11.5.10 Transfer the sample to a glass beaker with 4 N HNO_3.

11.5.11 Wash down the beaker and lid with small portions of 4 N HNO_3 and add to beaker.

11.5.12 Evaporate to dryness on hot plate. Do not allow sample to splatter. Remove from heat and allow to cool to room temperature.

11.5.13 Add 10 mL of concentrated nitric acid.

11.5.14 Evaporate to dryness on a hot plate. Do not allow sample to splatter.

11.5.15 Remove from heat and allow to cool to room temperature.

11.5.16 Add 10 mL of 4 N nitric acid. Heat to dissolve and then to reduce volume to approximately 5-7 mL.

11.5.17 Transfer sample to a ridged stainless steel planchet.

11.5.18 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet

11.5.19 Evaporate to dryness on a hot plate, do not allow the sample to splatter.

11.5.20 Remove sample from hot plate.

11.5.21 If sample appears hygroscopic, dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours. If not hygroscopic proceed to step 11.5.22.

11.5.22 Cool planchets in a desiccator for a minimum of 30 minutes.

11.5.23 Weigh the cooled planchets and record final weight.

11.5.23.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.5.23.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.5.23.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).

11.5.24 Store dry sample planchets in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.6 Filter Samples

11.6.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.

11.6.2 If the filter is less than 2" diameter, secure the air filter in a stainless steel planchet with double-sided cellophane tape such that no portion of filter extends above the lip of the planchet. Then proceed to step 11.6.20.

11.6.3 If the filter is 2" diameter, the sample can be placed directly in the dectector.

11.6.4 If the filter is greater than 2" diameter, digest or leach the sample per ST-RC-0004 for shared filters. Prepare a method blank and LCS from blank filters, spiked as per 9.3.3, which are digested in the same manner as the samples.

11.6.5 Shake the digested sample thoroughly. Measure a volume of sample into an appropriately sized teflon beaker. Record volume of sample used.

11.6.6 Add 10 mL of 16 N nitric acid.

11.6.7 Evaporate to dryness on a warm hot plate. Do not allow the sample to splatter.

11.6.8 Remove from heat and allow to cool to room temperature.

11.6.9 Add 10 mL of 16 N nitric acid.

11.6.10 Evaporate to dryness on a warm hot plate, do not allow the sample to splatter.

11.6.11 Remove from heat and allow to cool to room temperature.

11.6.12 Add 10 mL of 4 N nitric acid.

11.6.13 Heat to dissolve and then to reduce volume to approximately 5-7 mL

11.6.14 Transfer the sample to a pre-weighed, stainless steel planchet.

11.6.15 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet.

11.6.16 Evaporate to dryness on a warm hot plate. Do not allow the sample to splatter.

11.6.17 If sample appears hygroscopic dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours. If not hygroscopic proceed to step 11.6.18.

11.6.18 Cool planchets in a desiccator for a minimum of 30 minutes.

11.6.19 Weigh the cooled planchets and record final weight.

11.6.19.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.6.19.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.6.19.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).

11.6.20 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.7 Solid and/or Soil Samples by Dry, Grind Sprinkle

11.7.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.

11.7.2 If the sample has already been prepared per ST-RC-0003, "Drying and Grinding of Soil and Solid Samples," proceed to step 11.7.8 for direct sample mounting.
11.7.3 Use table salt for the blank and a soil standard reference material, e.g. NIST Traceable Rocky Flats Soil, for the LCS. Prepare in the same fashion as the samples.
11.7.4 Remove an aliquot (typically 1 - 5 g.) with a spatula and place into a clean, labeled aluminum weighing pan.
11.7.5 Place sample on a hot plate or in a drying oven at approximately 105° C and evaporate any moisture.
11.7.6 When dry, remove from hot plate or oven and allow the sample to cool.
11.7.7 If necessary, using a metal spatula, reduce the solid sample to a fine particle size.
11.7.8 Use double sided tape to secure the self-adhesive dots (adhesive side up) to a flat stainless steel planchet. Self adhesive label dots are used to hold finely divided solid material uniformly for gross alpha and/or beta analysis. Weigh and record the prepared planchet.
11.7.9 Distribute the sample evenly in the stainless steel planchet.
11.7.10 Record final weight. The target mass is 40-100 mg.
11.7.10.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.7.10.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.7.10.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.7.11 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.8 Solid and/or Soil Samples by total dissolution.
11.8.1 Initiate sample preparation sheet.
11.8.2 Weigh 1.0g sample into a 50 mL beaker and record weight.
11.8.3 Place in oven at 600° and allow to muffle for four hours. Allow to cool.
11.8.4 Transfer to digestion tube using 4 M HNO_3.
11.8.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
11.8.6 Digest in mod block at > 110°C for approximately four hours or until dry.
11.8.7 Carefully add 5 mL concnetrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
11.8.8 Digest in mod block at > 110°C for approximately four hours or until dry.
11.8.9 Add 10 mL HNO_3 and digest in mod block at >110°C for approximately 4 hours or until dry.
11.8.10 Reflux sample with 10 mL 4M HNO_3 for 20 minutes using a watchglass over digestion vessel.
11.8.11 Bring up to 20 mL with 4M HNO_3 in the digestion vessel.
11.8.12 Transfer 1 mL of sample to a tared planchet and cook to dryness.
11.8.13 Cool in descicator for 30 minutes.
11.8.14 Reweigh the planchet to determine the mass of 1 mL.
11.8.15 Determine the total amount of sample needed to reach the target mass of 100 mg on the planchet.
11.8.16 Transfer amount of sample to a 250 mL beaker.
11.8.17 Prepare blank, MS and LCS.
11.8.18 Add 10 mL of HNO_3 and cook to dryness. Allow to cool.
11.8.19 Add 10 mL of 4 N nitric acid.

11.8.20 Heat on hot plate to dissolve sample residue and then to reduce volume to approximately 5-7 mL.
11.8.21 Transfer the sample to a ridged stainless steel planchet.
11.8.22 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet.
11.8.23 Evaporate to dryness on a warm hot plate. Do not allow liquid to splatter.
11.8.24 Remove sample from hot plate.
11.8.24.1 If sample appears hygroscopic, dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours.
11.8.25 Weigh the cooled planchets and record final weight.
11.8.25.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.8.25.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.8.25.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.8.26 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.9 Reprocessing planchets which are over the weight limit.
11.9.1 Rinse residue from planchet with 4 N HNO_3 into a beaker. Add 4 N HNO_3 to planchet and heat if necessary to complete the transfer.
11.9.2 Redissolve the residue into 4 N HNO_3. Dilute the sample to a known volume.
11.9.3 Remove an aliquot which will keep the residue weight under the limit (100 mg) and transfer to the pre-weighed planchet. Record information on sample worksheet.
11.9.4 Evaporate to dryness on a warm hot plate so that the sample does not boil.
11.9.5 Remove sample from hot plate. Allow to cool.
11.9.6 Weigh the cooled planchet and record final weight.
11.9.7 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analytical SOP.
12.2 To calculate the aqueous sample volume required (ml), use the following equation:

$$volumerequired(mL) = \frac{targetnet\,residueweight(mg) * initialaliquotvolume(mL)}{initialaliquotnet\,residueweight(mg)}$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analytical SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

17.0 REFERENCES

17.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," Method 900.0, August, 1980.

17.2 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Method 9310, Rev. 0, September, 1986.

17.3 DOE Method RP-710, "Laboratory Method for Gross Alpha and Beta Activity Determination, 1997

17.4 TestAmerica St. Louis Laboratory Quality Assurance Manual (ST-QAM)

17.5 Corporate Environmental Health and Safety Manual (CW-E-M-001) and Facility addendum.

17.6 Associated SOPs

17.6.1 ST-PM-0002, Sample Receipt and Chain of Custody
17.6.2 ST-RC-0002, Preparation of Stainless Steel Planchets for Radiochemistry Analyses.
17.6.3 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
17.6.4 ST-RC-0004, Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis
17.6.5 ST-RC-0021, Gross Alpha Radiation in Water Using Coprecipitation
17.6.6 ST-RD-0403, Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System
17.6.7 ST-RC-5006, Decontamination of Laboratory Glassware. Labware and Equipment
17.6.8 ST-QA-0002, Standards and Reagent Preparation
17.6.9 ST-QA-0005, ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.6.10 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated section 9.2.7: added leached analyses use of a method blank that is comprised of leaching acid.
19.2 Replaced piptte with pre-weighted beaker to measure sample aliquot in section 11.3.3.

19.3 Rev 14:
19.3.1 Updated the total dissolution procedure for solid/soil samples in Section 11.8.

19.4 Revision 15:
19.4.1 Added required pH checking for all aqueous samples prior to analysis in section 8.3.
19.5 Revision 16:
19.5.1 Added reference to DOE Method RP-710 to Sections 1 and 17

19.6 Revision 17:
19.6.1 Updated section 15.
19.6.2 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.
19.6.3 Removed references to ‘Clouseau” and “Quantims”, replaced with LIMS.
19.6.4 Updated method requirements for Air Filter samples in section 2.0.
19.6.5 Updated supplies in section 6.0.
19.6.6 Updated reagents and standards in section 7.0.
19.6.7 Replaced the use of a porcelain crucible with a beaker throughout section 11.0.
19.7 Rev.18:
19.7.1 Section 8, removed sample hold time
19.7.2 Section 11.3.1, added GAB solid screen forrn Rad-0052
19.7.3 Section 11.1.10, added GAB solid screen form Rad-0052
19.7.4 Grammatical errors fixed throughout

Title: PREPARATION OF SAMPLES FOR GAMMA SPECTROSCOPY [EPA 901.1 and DOE GA-01-R]

Approvals (Signature/Date):

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This SOP was previously identified as SOP No. ST-RC-0025 Rev. 15

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this SOP is to provide detailed instructions for the preparation of samples which require gamma spectroscopy analysis.

1.2 This SOP describes methods for the preparation of samples of liquid, soil, vegetation, air filter, and core matrices prior to gamma spectroscopy analysis.

1.3 This SOP is based on EPA Method 901.1 and DOE Method GA-01-R.

1.4 The laboratory target analytes supported by this method, the reporting limits, and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Samples are transferred to a standard geometry container for counting on the gamma detectors. High purity germanium (HPGe) gamma detectors are used to detect isotopes with gamma ray energies between 40 and 2000 KeV. Activity concentration is determined using commercially available gamma spectral analysis software. A sample matrix which can be mounted in one of the standard geometries may be analyzed for any of the isotopes included in the radionuclide reference library. Detection limits may be affected by the sample size. Gamma photon energies not identified in the reference library may be identified and evaluated manually.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Replicate Analyses – Two or more analysis of the same sample whose independent measurements are used to determine the precision of equipments analytical procedure.

4.0 INTERFERENCES

4.1 Gamma energy emissions identified with scientifically measured probability by some radionuclides are documented by multiple sources. There are some discrepancies between reference sources and attempts are made to evaluate the reference data used in spectral analysis. Gamma emissions at discreet energy and probability are used to identify and quantify specific radionuclides in the sample. Gamma emissions which are completely absorbed by an HPGe detector form photo peaks which are used for identification and quantification of gamma emitting radionuclides. When two or more nuclides emit similar gamma energy the photo peaks cannot be resolved without using complex algorithms. These photo peaks in close proximity can interfere with the identification or quantification of a radionuclide. Knowing this the nuclide reference library, computer software and analyst training are used to minimize the possibility of interference and misidentification. It is not possible to eliminate all interferences and misidentification.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of

the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 **Wear Kevlar or MAPA Blue-Grip gloves when using knives or sharp articles.**

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. ***NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.*** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Nitric acid can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1- Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance, top loader

6.2 Blender

6.3 Food chopper/grinder

6.4 Knives appropriate for food preparation

6.5 Graduated cylinder

6.6 Filter disk, 47 millimeter diameter

6.7 Plastic Tape

6.8 Marinelli beakers of various sizes (500 mL and 1000 mL)

6.9 Petri dishes, 2-inch diameter

6.10 Can Sealer

6.11 Cans and lids (commonly referred to as tuna cans)

6.12 8 oz, straight sided polypropylene jars or equivalent; (used for 25 mL and 100 mL geometries)

6.13 Teflon® or glass beakers (250 mL, 400 mL)

6.14 Disposable digestion vessels

6.15 Muffle furnace (programmable)

6.16 TEXPEN®

6.17 Teflon® beaker covers

6.18 Watch glasses

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 DI Water obtained from the Milli-Q® unit.

7.3 Nitric acid (16 N HNO_3) concentrated

7.3.1 Nitric acid (4 N HNO_3) – to an appropriately sized bottle containing 1500 mL of DI water; add 500 mL of 16 N HNO_3.

7.4 Hydrochloric acid (12 N HCL) – concentrated, 37.2%

7.5 Hydrofluoric acid (HF 48.52%) – concentrated

7.6 Radiacwash™ solution 10% - add 100 mL of Radiac to 1 L of water

7.7 Bleach solution 10% - add 100 mL of bleach to 1 L of water

7.8 Sodium Sulfate

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2, unless I-129 or I-131 is requested. Samples collected for I-129 or I-131 analysis are ***not preserved***.

8.3.1 The pH of aqueous samples is checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis

8.3.1.1 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior to analysis.

8.4 Milk samples are not chemically preserved.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (i.e. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Replicate.

9.1.3.1 A Sample Duplicate may be performed at the request of the client. See client requirements.

9.2 **Method Blank**

9.2.1 For **Water** and **Liquid** analyses, the method blank is comprised of DI water.

9.2.2 For **Soil** and **Solids** analyses, the method blank is comprised of sodium sulfate.

9.2.3 For Filter analyses, the method blank is comprised of a Petri dish.

9.2.4 A Method Blank must be prepared with every sample batch.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 The LCS is a purchased sealed source standard in the prescribed geometry for the sample analysis.

9.3.3 An LCS must be prepared with every sample batch.

9.4 **Sample Duplicate**

9.4.1 A sample duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision (a replicate analysis of the original sample counted on a different detector will be performed as the duplicate).

9.5 **Procedural Variations/ Nonconformance and Corrective Action**

9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6 **Decontamination of Tuna Can Sealer**

9.6.1 The sealer must be wiped down with 10% Radiacwash™ solution daily

9.6.1.1 Analyst must record the information in the daily logbook..

9.6.2 Sealer will be monitored for contamination as part of the monthly contamination survey, as per SOP ST-RP-0032

10.0 CALIBRATION AND STANDARDIZATION

10.1 The balance must be calibrated in accordance with ST-QA-0005.

10.2 For Gamma Spectroscopy calibration requirements, see ST-RD-0102.

11.0 PROCEDURE

11.1 Liquid Sample Preparation

11.1.1 Liquid samples shall be prepared as a 25 mL, 100 mL, 500 mL, or 1000 mL geometry.

11.1.2 Determine the proper geometry.

11.1.2.1 The volume of sample used depends on the amount required to meet the detection limits, the volume of sample supplied by the client, and whether the sample has very high activity. The sample volume may be reduced for high activity samples due to detector dead time considerations. Consult the count room supervisor or radiochemistry technical director, if the sample has high activity which may require such consideration.

11.1.3 Label the top of the container with the sample Id

11.1.4 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.

11.1.5 Write sample information (i.e. ID #) on the container.

11.1.6 Measure the required sample volume (25, 100, 500 or 1000 mL) by comparing the sample to the reference container

11.1.6.1 Reference containers are pre-made geometries comprised of DI water measured volumetrically.

11.1.6.2 If the client does not provide sufficient sample, and the sample is near a larger geometry, rather than reducing the volume significantly it may be preferable to dilute an aqueous sample with DI water to the correct volume in order to achieve a lower MDC. Consultant Supervisor/Manager to determine which action is preferable.

11.1.6.2.1 If the sample is diluted the undiluted volume is recorded as the sample volume. The dilution is only for fitting the calibrated geometry.

11.1.7 Place the lid securely on the container.

11.1.7.1 Remove excess air from Marinelli.

11.1.7.2 If the density is suspected to be greater than 1.2 g/ mL or less than 0.98 g/mL, generate a NCM. To determine the density use form RAD-0075_Density.xls (include form with batch paper work if utilized) Attachment 1

11.1.7.2.1 Form directory: \\slsvr01\QA\FORMS\ST-LOUIS\RAD

11.1.8 Seal the lid using plastic electrical tape. Marinelli beakers are prone to leaking liquids; the tape is tightly wrapped around the lid and the beaker in three layers each overlapping the previous layer with half the width of the tape. Make sure there are no creases in the tape which will form a channel for leakage.

11.1.9 Inspect for leakage.

11.1.10 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.2 Soil Sample Preparation

11.2.1 Soil samples for I-129 or I-131 analysis **are not dried and ground** but rather inserted into an appropriate calibrated geometry. Proceed to step 11.2.3.

11.2.2 Soil samples, which do not require I-129 or I-131 analyses, are prepared in accordance with SOP ST-RC-0003 or ST-RC-0014.

11.2.3 Soil samples shall be prepared as 200 mL sealed (tuna) can, 100 mL, or 25 mL, or 500 mL Marinelli (marnsoil) geometry based on the amount of available sample. In both the tuna can and marnsoil geometries, the soil should nearly fill the container.

11.2.3.1 For I-129 analysis only a 25 mL or 100 mL straight sided poly jar geometry may be used (check with count room analyst on which geometry I-129/I-131 is calibrated for and prep the sample using a matching geometry).

11.2.4 Write sample information (i.e. ID #) on the sample container.

11.2.5 Pre-weigh the empty container (tare weight) and record weight in TALS and on the container lid.

11.2.6 Fill the container with the appropriate amount of sample as described below.

11.2.6.1 Fill tuna cans to the ridge mark with sample. If there is insufficient sample to fill the can to the ridge, reduce geometry size.

11.2.6.2 Fill 100 mL geometry to the level as denoted on the reference container. If there is insufficient sample to fill, reduce the geometry size.

11.2.6.2.1 A 100 mL "reference" bottle is marked with the appropriate fill level.

11.2.6.3 Fill 25 mL geometry to the level as denoted on the reference bottle. If there is insufficient sample to fill the 25 mL geometry, write a NCM stating insufficient sample provided for routine analysis.

11.2.6.3.1 A 25 mL "reference" container is marked with the appropriate fill level.

11.2.6.4 Fill 500 mL Marinelli beakers to the ridge mark just below the lid with sample. If there is insufficient sample to fill the marnsoil to the ridge, reduce geometry size.

11.2.7 Close the sample container securely.

11.2.8 Seal the container with plastic electrical tape. For tuna cans, seal with can sealer, wipe samples clean with paper towel and DI water.

11.2.9 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of container plus sample, minus the tare weight of the empty container).

11.2.10 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.3 Vegetation Sample Preparation (No digestion)

11.3.1 Vegetation samples may be prepared in an appropriate calibrated geometry counted directly as dried and chopped matrix or green unprocessed matrix (if directed to do so by the client or if I-131 or I-129 is to be reported).

11.3.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by client or Project Manager.

11.3.1.1.1 Vegetation samples **for I-129 or I-131 analysis are not dried** but rather inserted into an appropriate calibrated geometry.

11.3.1.1.2 Dry weight can be determined on sample(s) not dried by using the percent moisture.

11.3.1.2 Consult the client requirements, client requirement memorandums or the Supervisor/Manager to determine proper sample handling.

11.3.2 The sample shall be counted in a 500 mL Marinelli, 100 mL, or 25 mL geometry. The container is filled to the appropriate level with the sample.

11.3.2.1 I-129 analysis uses only a 25 mL or 100 mL straight sided poly jar geometry (check with count room analyst on which geometry I-129/I-131 is calibrated for and prep the sample using a matching geometry).

11.3.3 Write sample information (i.e. ID #) on the container.

11.3.4 Pre-weigh the empty container and record weight in TALS and on lid of container.

11.3.5 Place sample in the tared container.

11.3.5.1 Compress the sample when filling a 500 mL Marinelli beaker

11.3.6 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of container plus sample minus the tare weight of the empty container).

11.3.6.1 Verify with the Project Manager if sample is to be reported on a wet or dry basis.

11.3.7 Close the sample container securely, seal with plastic electrical tape

11.3.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.4 Vegetation Sample Preparation (with digestion)

11.4.1 Vegetation samples may be digested and then placed into a appropriate calibrated geometry and counted as a liquid matrix.

11.4.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by the client or project manager.

11.4.1.1.1 **Iodine isotopes (e.g. I-125, I-129 or I-131) cannot be prepared using digestion technique** due to volatility.

11.4.1.2 Consult the client requirements and Supervisor/Manager to determine proper handling.

11.4.2 Label an appropriate sized beaker with a TEXPEN® to ensure identification post muffling

11.4.3 Transfer sample into beaker and record the weight on the gamma worksheet

11.4.3.1 Check with the Project Manager to determine if sample is to be reported on a wet or dry basis.

11.4.4 Cover sample with 8 N HNO_3 and allow to sit overnight.

11.4.5 Place beaker on hot plate and gently heat so that sample does not splatter. Periodically stir sample

11.4.6 Wash down sides of the beaker with small amounts of 30% H_2O_2 and small amounts of 8M HNO3. Continue this process periodically as sample digests on hot plate.

11.4.7 Cook sample down to dryness.

11.4.7.1 Upon visual inspection if sample is orange in color, cover sample with 16M HNO_3 and continue to wet ash with 30% H_2O_2.

11.4.7.2 Repeat 11.4.7 and 11.4.7.1 until sample is light yellow or white in color.

11.4.8 Cover beaker with a watch glass and place into the muffle oven.

11.4.9 Heat at 600° C for a minimum of 4 hours to reduce the sample

11.4.10 Allow beakers to cool to room temperature before removing from the oven.

11.4.10.1 The sample may be wet ashed again if yellow color remains.

11.4.11 Reflux samples with enough of 4 N HNO_3 to cover sample for approximately 30 minutes, using a watch glass to cover the beaker.

11.4.12 Quantitatively transfer sample to a labeled digestion vessel or Teflon® beaker with a minimal amount of 4 N HNO_3.

11.4.13 Add 5 mL of 16 N HNO_3, 5 mL of 12 N HCl and 10 mL of concentrated HF

11.4.14 Bring sample to dryness

11.4.14.1 If using a digestion vessel, place in MOD Block

11.4.14.2 If using a Teflon® beaker, heat on a hotplate

11.4.15 Repeat steps 11.4.10 and 11.4.11

NOTE: The amount of acid will vary depending on the sample aliquot. When increasing the aliquot, increased acid will be required

11.4.16 Reflux the sample in 10 to 20 mL of 4 N HNO_3. Allow to reflux with a watch glass over the beaker for approximately 30 minutes to ensure samples goes into solution.

11.4.16.1 If sample does not go into solution additional digestions may be required (see Supervisor/Manager/Technical Director for guidance)

11.4.17 Label appropriate size container (25 or 100 mL)

11.4.18 Quantitatively transfer sample to container by rinsing with minimal amount of 4 N HNO_3

11.4.19 Bring to volume to match the reference container

11.4.20 Close the sample container lid securely and seal with plastic electrical tape.
11.4.21 Inspect for leakage.
11.4.22 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.5 Air Filters/Swipes (no digestion)
11.5.1 Air filters may be counted as single filters or as composite filters.
11.5.1.1 Consult client requirements and Supervisor/Manager for instruction.
11.5.1.2 Air filters are reported as pCi/sample or pCi/g. The weight to the air filter is required if the reporting units is pCi/g.
11.5.2 Write sample information (i.e. ID#) on Petri dish.
11.5.3 Filters with reporting units of
11.5.3.1 pCi/g proceed to 11.5.4
11.5.3.2 pCi/sample proceed to 11.5.5
11.5.4 Pre-weigh empty Petri dish, record the weight in TALS and then on the lid of the Petri dish.
11.5.5 Load air filter(s) directly into Petri dish
11.5.6 Place lid on Petri dish, for pCi/g record the weight of the sample (container and sample weight, minus container weight) in TALS.
11.5.7 Secure the Petri dish lid with plastic electrical tape
11.5.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.6 Air Filters/Swipes (with digestion)
11.6.1 Air filters are digested and counted as a liquid.
11.6.2 Label a 250 mL beaker with a TEXPEN® to ensure identification post muffling.
11.6.3 Place sample into beaker
11.6.4 Place sample beaker in muffle oven and cover with a watch glass.
11.6.5 Ramp air filters in muffle oven
11.6.5.1 Refer to ST-RC-004 for ramping settings
11.6.6 Allow beakers to cool to room temperature
11.6.7 Digest filter in accordance with sections 11.4.9 to 11.4.12
11.6.8 Label appropriate size container (25 to 100 mL)
11.6.9 Transfer sample to container with minimal amount of 4 N HNO_3.
11.6.10 Bring to volume using reference container as a guide
11.6.11 Place lid securely on the container
11.6.12 Seal the lid using plastic electrical tape
11.6.13 Inspect for leakage.
11.6.14 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.7 Core Samples
11.7.1 To obtain sample, cut Shelby tube or sample container into two pieces.
11.7.1.1 Using a rigid pipe cutter cut the tube completely through.
11.7.1.2 Using a wire saw, cut through the sample.
11.7.1.3 Cuts should be made at 2 inch intervals.
11.7.1.4 Remove sample from every other sliced section of the Shelby tube.
11.7.1.5 Dry and grind the sample as described in SOP ST-RC-0003.
11.7.2 Aliquot 500 g of sample
11.7.2.1 If less than 500 g of sample is available, contact Supervisor/Manager for instruction.
11.7.2.2 Soil samples shall be prepared as 200 mL sealed (tuna) can, 100 mL, or 25 mL, or 500 mL Marinelli (marnsoil) geometry based on the amount of available sample.

In both the tuna can and marnsoil geometries, the soil should nearly fill the container.

11.7.3 Pre-weigh the empty container and record the weight on the container lid and in TALS
11.7.4 Writer sample information (i.e. ID#) on the sample container
11.7.5 Place the dried sample into the container for counting.
11.7.6 Weigh and record sample weight in TALS .
11.7.7 Secure the lid on the container with plastic electrical tape.
11.7.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.8 Food: vegetables, produce, grain or animal feed:

11.8.1 Vegetables, produce and grain samples may be prepared in a 500 mL Marinelli beaker or 1 L Marinelli beaker geometry depending on the requested reporting limit. These matrices are counted directly as whole grain, chopped or blended produce or vegetable matrices **without drying** unless directed by the client to dry the matrix.
11.8.1.1 Consult the client requirements and Supervisor/Manager for instruction.
11.8.2 For vegetables and produce, prepare the sample by chopping with a knife on a cutting board or using a food processor.
11.8.3 Write sample information (i.e. ID #) on the appropriate container.
11.8.4 Pre-weigh the empty container and record weight on container and in TALS.
11.8.5 Place processed sample in the pre-weighed container.
11.8.6 Compress the sample when filling a 500 mL Marinelli beaker
11.8.7 Weigh sample and record the weight in TALS as WET weight in grams.
11.8.7.1 If the sample is dried, record the DRY weight.
11.8.8 Close the sample container securely, seal with plastic electrical tape.
11.8.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.9 Food: meat and fish:

11.9.1 Meat and fish may be prepared in a 500 mL Marinelli beaker or 1 L Marinelli beaker geometry depending on the requested reporting limit. These matrices are counted directly **without drying.**
11.9.1.1 Consult the client requirements and Supervisor/Manager for instruction.
11.9.2 For meat and edible portions of fish, prepare the sample by chopping with a knife on a cutting board.
11.9.2.1 Fish sample are to be filleted prior to chopping.
11.9.2.2 For analysis of fish **when the whole fish is required to be analyzed**, remove the head with a knife and cut the fish into pieces of appropriate size to easily fit into the Marinelli beaker without air voids. Place the heads in the main portion of the Marinelli and surround it with pieces to eliminate air voids or spaces.
11.9.3 Write sample information (i.e. ID #) on the container.
11.9.4 Pre-weigh the empty container and record weight on container and in TALS.
11.9.5 Place processed sample in the pre-weighed container.
11.9.6 Compress the sample evacuating any space in the geometry when filling a Marinelli beaker
11.9.7 Weigh sample and record the weight in TALS as WET weight in grams.
11.9.8 Close the sample container securely, seal with plastic electrical tape.
11.9.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.10 Store unused portions of sample in appropriately sized poly containers. Food and Vegetation samples are to be refrigerated.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 There are no calculations pertaining to this sample preparation procedure.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

12.3 Percent Moisture: Mass of original sample minus mass of dried sample divided by mass of dried sample.

12.3.1 $$p = \frac{W - D}{D}$$

p = fraction of total evaporable moisture content of sample
W = mass of the original sample
D = mass of dried sample

12.4 Density: Sample weight divided by the volume of said sample weight

12.4.1 $$d = \frac{m}{v}$$

d = density
m = mass
v = volume

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM.

14.3 Training Qualification
- 14.3.1 The Supervisor/Manager has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in ST-QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where feasible, technological changes have been implemented minimizing the potential for pollution to the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method
The following waste streams are produced when this method is carried out.

16.2.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method EPA 901.1.

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, GA-01-R.

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revision.

17.5 Associated SOPs

17.5.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
17.5.2 ST-RC-0004, Preparation of Soil, Sludge, and Filter Paper Samples for Radiochemical Analysis
17.5.3 ST-RC-0014, Bulk Drying and Grinding of Soil and Solid Samples
17.5.4 ST-RD-0102, Gamma Spectroscopy Analysis
17.5.5 ST-RP-0032, Instrumentation and Surveillance
17.5.6 ST-PM-0002, Sample Receipt and Chain of Custody
17.5.7 ST-QA-0002, Standard and Reagent Preparation
17.5.8 ST-QA-0036, Non-conformance Memorandum (NCM) process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 No Changes, Annual Review

19.2 Rev 11:

- 19.2.1 Inserted instructions regarding requirements for checking the pH of samples upon receipt and or prior to analysis in section 8.3.1.
- 19.2.2 Updated when sample ID's should be written on the container in section 11.0.
- 19.2.3 Inserted instructions for generating container ID labels throughout section 11.0.
- 19.2.4 Updated when weighing and recording sample weights/mass should be documented in gamma worksheet in section 11.0.
- 19.2.5 Added instructions for properly cleaning tuna cans before storage in section 11.2.9.

19.3 Rev 12:

- 19.3.1 Extensive revision to entire procedure
- 19.3.2 Addition of attachment 1

19.4 Rev 13:

- 19.4.1 Added sodium sulfate to section 7.0.
- 19.4.2 Updated section 9.2 regarding the matrix of method blanks.

19.5 Rev 14:

- 19.5.1 Updated vegetation sample with and without digestion throughout section 11.3 and 11.4.

19.6 Rev. 15: (8/30/2013)

- 19.6.1 Grammatical corrections and removal of references to QuantIMS through out
- 19.6.2 Section 3, added definition of replicate analyses
- 19.6.3 Section 8, removal of 180 day holding time
- 19.6.4 Section 9, explained that replicate will be used as duplicate for this procedure
- 19.6.5 Section 9.6, added "record in daily logbook"
- 19.6.6 Deleted record tare weight on lid in section 11.1.2
- 19.6.7 Deleted record weight of sample (container and sample weight minus container weight) on lid in section 11.1.8 , 11.2.9, 11.3.6
- 19.6.8 Added record weight in TALS throughout SOP
- 19.6.9 Added print proper paperwork throughout SOP

19.7 Rev. 16: (1/16/2015)

- 19.7.1 Grammatical corrections through out
- 19.7.2 Updated section 11.0
- 19.7.3 Added SOP references to Section 17: ST-RC-0014, St-RP-0032

Attachment 1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

DENSITY CORRECTION

Date: _____ Digi Tube Lot #: _____

Analyst: _____ Balance ID: _____

	Sample ID:	Container Wt (g)	Sample Volume (mL)	Sample + Container Wt (g)	Density (g/mL)	Original Sample Aliquot	Adjust Sample Aliquot
1							
2							
3							
4							
5							
6							

Instructions:

Record the weight of a Class A digi tube in "Container Wt(g)"; record the volume of the sample added to the digit tube in "Sample Volume (mL)"; record the sample and digi tube in "Sample + Container Wt(g)." "Original Sample Aliquot" is the full sample size used for the actual analysis.

This density is to be used for sample aliquot correction when a sample is suspected of density less than or greater than 1. Include this document with batch paperwork.

Review is to be completed by a peer (someone other than the analyst handling the samples).

Reviewed by: _____ Date: _____

\\slsvr01\QA\FORMS\ST-LOUIS RAD-0075 REV (0)

Title: THE DETERMINATION OF TRITIUM IN WATER (AND OTHER FLUIDS), SOIL, SILICA GELS, AND GRAPHITE

Approvals (Signature/Date):

Sarah Bernsen 8/6/14 Sarah Bernsen Date Radiochemistry Prep Supervisor	Michael Ridenhower 8/6/14 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 8-6-14 Marti Ward Date Quality Assurance Manager	Elaine Wild 8/7/14 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0030 Rev. 16

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure for the preparation of samples for the determination of tritium in water (and other fluids), soil and silica gels.

1.2 This procedure is based on EPA Method 906.0 and DOE Method H3-04-RC.

1.3 The instrument analysis is described in SOP ST-RD-0302.

1.4 Steam Distillation is applicable to silica gels, soils (water added) and ground, surface, drinking or other types of water samples, where sufficient volume exits to perform steam distillation. The sample is free from quenching agents that would prevent the use of the Liquid Scintillation Counter (LSC) generated quench curve for efficiency determination.

1.5 Direct Method is applicable to those liquids where distillation is impractical, yet the LSC generated quench curve efficiency would still be appropriate (body fluids, oils, oil-water mixtures). **Direct Counting is NOT acceptable for Drinking Water samples.**

1.6 Standard Addition is applicable to either of the two previous methods, when excessive quenching prevents a reasonable determination of counting efficiency. By manipulation of counting times, this procedure is appropriate for high activity or environmental samples. **This procedure is not acceptable for Drinking Water samples.**

1.7 The reporting limits, method detectable activities (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP provides detailed instructions for the preparation of samples for the determination of tritium in water (and other fluids), soil and silica gels.

2.1.1 Steam Distillation - an aliquot of unpreserved water is steam distilled, and the condensate collected. Scintillation cocktail is added and following a suitable period of dark adaptation, the sample is counted in the LSC. Using a predetermined quench curve, a counting efficiency is assigned, and results are calculated.

2.1.2 Direct Method - a small volume of sample is transferred to a counting vial, cocktail is added, and the sample is counted following dark adaptation. A counting efficiency is assigned and results are calculated. **This is not used for Drinking Water samples.**

2.1.3 Standard Addition - a sample observed to exhibit excessive quenching by previous counting is spiked with a known amount of tritium. The sample is recounted, and the counting efficiency factor is accurately determined. This factor is then applied to the original counting data and results are calculated. **This is not used for Drinking Water samples.**

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

3.2 LSV – Liquid Scintillation Vial

4.0 INTERFERENCES

4.1 Tritium existing in background water is an interference. Slightly elevated levels of tritium are present in surface waters, therefore it is preferable to use water from a deep well source.

4.2 Most interferences can be overcome using one of the three methods described in section two. Distillation would remove most interferences from the sample.

4.3 The use of plastic vials may cause buildup of static charge and give erratic results. The instruments contain anti-static controllers but the additional preventative measure of wiping the vial with an anti static cloth aids in prevention of erratic static buildup. Wiping the plastic vials with an anti static dryer sheet works well to prevent the buildup and discharge of static.

4.4 Glass Liquid Scintillation Vials may contain low amounts of potassium which includes the beta emitting natural isotope, K-40. When glass LSC vials are used, they should be made from low potassium glass.

4.5 Nitrogen will generate chemical quench, samples should not be preserved with nitric acid.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

5.3.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive Poison	2 ppm (TWA)	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Potassium Permanganate	Oxidizer	5 Mg/M^3 (Mn Compounds) (Ceiling)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time weighted average			
STEL – Short term exposure limit			
Ceiling – At no time should this exposure limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Steam distillation setup, with heating mantles, flasks, condensers and Barrett trap

6.2 Liquid scintillation vials (LSV) - glass or plastic, as appropriate, 20-mL volume

6.3 Nalgene bottles or equivalent, 250-mL volume with screw cap

6.4 Centrifuge tubes , poly 50-mL with cap.

6.5 Calibrated pipettes - Eppendorf or equivalent

6.6 Adjustable dispenser for liquid scintillation cocktail, range; 1 to 20-mL

6.7 Boiling Chips

6.8 Analytical Balance

6.9 250-mL, 500-mL, and 1-L flask, round and flat bottom

6.10 20-mL Barett trap

6.11 Silica sand

6.12 Anti-static “dryer” sheets

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI Water, obtained from the Milli-Q unit.

7.3 Background water (dead water) – deionized/reagent water with a tritium content at or below ambient environmental level, (maximum of 400 pCi/L). Deep well water is preferable when it is determined to have a lower tritium concentration than the deionized/reagent water. The tritium of this water should be well characterized.

7.3.1 Dead water is distilled on the tritium wall to create background water. Once the background water has been collected, it needs to be verified. Create a manual batch in Rad Capture and prepare 4 vials. 1 vial will be comprised of 10 mL of your current background water. The other 3 vials will be comprised of 10 mL of the new background water. Add 10 mL of Ultima Gold LLT to each sample. Shake the vials and wipe them down with a dryer sheet prior to turning them into the count room.

7.3.2 Once the background water passes verification (< 400 pCi/L), then a reagent label will be created in TALS. The verification paperwork will also need to be attached to the reagent document tab in TALS.

7.4 Tritium Solution, standard 400 – 100 dpm

7.5 Sodium Hydroxide pellets

7.5.1 10 N Sodium Hydroxide - Dissolve 100 g sodium hydroxide in 200 mL deionized water. Cool to room temperature and dilute to 250 mL with DI water.

7.6 0.5 M Potassium Permanganate - Dissolve 39.5 g potassium permanganate in 200 mL deionized water. Dilute with deionized water brought up to 500 mL.

7.7 Liquid scintillation cocktail - Packard Ultima Gold LLT (Low Level Tritium) or use the stated scintillation solution or its equivalent.

7.7.1 Each new lot of cocktail needs to be verified to ensure there are no interfences.

7.7.1.1 Prepare ICV as detailed in SOP ST-RD-0302 vials and submit to count room with proper paperwork. Apply acceptance criteria from ST-RD-0302 (± 10% of the known value).

7.8 Methanol

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples should not be preserved.

8.2.1 The pH will be verified prior to analysis,

8.3 The sample should be collected in a glass container.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), a Matrix Spike (MS) and Sample Duplicate (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water.

9.2.4 For Soil analyses, the method blank is comprised of sand.

9.2.5 For Oil analyses, the method blank is comprised of vaccuum pump oil.

9.2.6 The method blank must be analyzed on a different condensor per batch. The condensor needs to be randomly selected prior to analysis and documented in logbook.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Tritium.

9.3.4 For Soil analyses, the LCS is comprised of sand fortified with Tritium.

9.3.5 For Oil analyses, the LCS is comprised of vaccuum pump oil fortified with Tritium.

9.3.6 The LCS must be analyzed on a different condensor per batch. The condensor needs to be randomly selected prior to analysis and documented in logbook.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.4.4 MS Sample for silica gels requires a post-distillation spike.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 A background sample is prepared and submitted for counting with each batch. The counts determined by the background sample are then subtracted from all samples counted in the batch including the blank.

10.1.1 A background sample consists of 10 mL "dead water" and 10 mL of ultima gold LLT.

10.2 Balance calibration must be checked each day of use. Refer to SOP ST-QA-0005 for details.

10.3 Pipette calibration must be checked each day of use. Refer to SOP ST-QA-0005 for details.

10.4 See analytical SOP for instrument calibration. ST-RD-0302

11.0 PROCEDURE

11.1 Steam Distillation (Water)

11.1.1 Rinse condensor lines with 5 mL of methanol and allow to dry completely (10-15 minutes)

11.1.2 Ensure that the sample container is capped tightly and shake well.

11.1.3 Initiate distillation paperwork.

11.1.4 Check that the pH $\geq$ 6.

11.1.4.1 If the pH is not $\geq$ 6, document the pH and generate an NCM.

11.1.4.2 Adjust the pH to $\geq$ 6 using 10.0 M NaOH (sodium hydroxide).

11.1.5 Transfer a 100 mL aliquot of sample to a round bottom flask.

11.1.6 Add 6 sodium hydroxide pellets and 1 mL of 0.5 N potassium permanganate.

11.1.7 Add boiling chips to prevent bumping in the distillation flask.

11.1.8 Attach the flask to the distillation setup and distill. For samples having a limited volume, 6 sodium hydroxide pellets and 0.1 g potassium permanganate crystals **may** be added instead of prepared solutions.

11.1.8.1 A Blank and an LCS consisting of a few boiling chips and 100 mL of DI water are prepared in the same manner as the samples. The LCS is spiked with a known amount of tritium.

11.1.8.2 A matrix spike and sample duplicate should also be prepared.

11.1.9 Attach the flask to the condensor and Barrett trap and begin distillation.

11.1.10 Collect the distillate in a Barrett Trap, discarding the first 5 mL. (Once heating mantles come to temperature, the distillation takes less than 1/2 hour to complete). **DO NOT collect the last 5 mL of distillate.**

11.1.11 Ensure that the flasks do not boil dry.
11.1.12 Collect the water distilled into a clean labeled 50-mL centrifuge tube.
11.1.13 Turn off the heating mantles and allow the samples to cool to room temperature.
11.1.14 Discard any remaining residue to waste.
11.1.15 Record the sample number on the cap of the liquid scintillation vial (LSV).
11.1.16 Collected sample must be at room temperature before an aliquot is taken.
11.1.17 Transfer 10 mL of distillate to a labeled LSV, using a pipet.
11.1.18 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.
11.1.19 Cap the vial and shake it to ensure complete mixing.
11.1.20 Inspect each LSV to ensure that it is free of dirt, grease, etc.
11.1.21 Using an anti static "dryer" sheet , wipe the sides of the LSV to reduce static.
11.1.22 Submit the samples to the counting lab for analysis.

11.2 Silica Gels

11.2.1 Rinse condensor line with 5 mL of methanol and allow to dry completely (10-15 minutes).
11.2.2 Tare a 1L round bottom flask, the sample should fill the inside bottom half of the flask.
11.2.3 Transfer the sample to the tared flask and record the sample weight.
11.2.4 Blank and LCS consist of 30 g of boiling chips and 40 mL of DI water. The LCS is spiked with a known amount of tritium.
11.2.5 Attach the flask to the distillation setup and distill the silica gels until the sample no longer produces water (approximately 6 hours). QC samples are distilled until at least 20 mL are collected. **Discard the first 5 mL of distillate.**
11.2.6 Collect all the water distilled into a clean labled 50 mL centrifuge tube.
11.2.7 Turn off the heating mantles and allow samples to cool to room temperature.
11.2.8 Weigh the sample to determine loss on drying. Record weight.
11.2.9 Return the dried silica gel to its original container.
11.2.10 Record the sample number on the cap of the LSV.
11.2.11 Transfer 10 mL of distillate to a liquid scintillation vial (LSV), using a pipet.
11.2.12 For the Matrix Spike, perform a post distillation spike on the distillate by removing 0.5 mL of distillate and adding a known amount of tritium.
11.2.13 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.
11.2.14 Cap the vial and shake it to ensure complete mixing.
11.2.15 Inspect each LSV to ensure that it is free of dirt, grease, etc.
11.2.16 Using an anti static "dryer" sheet , wipe the sides of the LSV to reduce static.
11.2.17 Submit the samples to the counting lab for analysis.

11.3 Solids (soil, filters/swipes if not direct counts)

11.3.1 Rinse condensor line with 5 mL of methanol and allow to dry completely (10-15 minutes).
11.3.2 Tare a 120 mL plastic specimen cup and add 30 g of soil to the cup.
11.3.3 Add 40 mL of DI water to each sample.
11.3.4 Prepare a Blank and LCS using 40 mL DI water and 30 g washed silica sand. LCS is spiked with a known amount of tritium.
11.3.5 Leach samples for at least 4 hours.
11.3.6 Transfer samples to 50 mL centrifuge tubes and centrifuge at 2000 rpm for at least 5 minutes.
11.3.6.1 If supernate is clear proceed to step 11.3.7.
11.3.6.2 If supernate is not clear filter using a 0.45 micron filter and then proceed to 11.3.6.
11.3.7 Transfer supernate to a 250-mL round bottom flask.
11.3.8 Attach the flask to the distillation setup and distill the samples until at least 20 mL are collected. QC samples are distilled until at least 20 mL are collected. **Discard the first 5 mL of distillate.**

11.3.9 Collect the water distilled into a clean labled 50 mL centrifuge tube.
11.3.10 Turn off the heating mantles and allow samples to cool.
11.3.11 Discard any remaining residue to waste.
11.3.12 Record the sample number on the cap of the LSV.
11.3.13 Transfer 10 mL of distillate to a liquid scintillation vial (LSV), using a pipet.
11.3.14 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.
11.3.15 Cap the vial and shake it to ensure complete mixing.
11.3.16 Inspect each LSV to ensure that it is free of dirt, grease, etc.
11.3.17 Using an anti static "dryer" sheet , wipe the sides of the LSV to reduce static.
11.3.18 Submit the samples to the counting lab for analysis.

11.4 Direct Method (**used for Oils and at client request for other matrices. Can NOT be used for Drinking Water**)
11.4.1 Record the sample number on the cap of the LSV.
11.4.2 Ensure that the sample is capped tightly and mix well.
11.4.3 Transfer an aliquot to an LSV.
11.4.3.1 Aliquot = 10 mL for water based samples
11.4.3.2 Aliquot = 1-2 mL for oil sample, depending on the color or transparency of the sample. A density test will have to be performed to get an accurate weight for the oil.
11.4.4 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.
11.4.4.1 For oils add 18-19 mL of cocktail (Insta Gel Plus) to equal 20 mL in the vial. Shake.
11.4.5 Cap the vial and shake it to ensure complete mixing.
11.4.6 Inspect each LSV to ensure that it is free of dirt, grease, etc.
11.4.7 Using an anti satic "dryer" sheet, wipe the side of the LSV to reduce static.
11.4.8 Record the sample number on the cap of the LSV.
11.4.9 Submit the samples to the cout room for analysis.

11.5 Internal Standardization (**Can NOT be used for Drinking Water)**
11.5.1 To the previously counted sample LSV, carefully open and add 1.0 mL of tritium SRM solution.
11.5.2 Replace the cap, shake well and wipe the LSV clean with a Kimwipe.
11.5.3 Record all data on the tritium data sheet.
11.5.4 Prepare one spiked sample, one blank, and a duplicate.
11.5.5 Submit the samples to the counting lab for analysis.

11.6 Graphite Samples
11.6.1 Using a cheese grater, grate enough of the sample to meet detection limits.
11.6.2 Leach for 12 hours in 40 mL of 20% KOH in a 120 mL plastic specimen cup.
11.6.3 Prepare a Blank and LCS by adding 100 mL of DI water to a 250-mL flask.
11.6.4 Spike the Matrix Spike and LCS with 0.5 mL of tritium.
11.6.5 Filter sample using 0.45 micron filter.
11.6.6 Add leachate to 250-mL round bottom flask and dilute to 100 mL with DI water.
11.6.7 Attach to distillation setup and distill.
11.6.8 Discard the first 5 mL of distillate to designated graphite waste container.
11.6.9 Collect at least 20 mL of distillate.
11.6.10 Allow distillate to cool to room temperature.
11.6.11 Aliquot 10 mL into a liquid scintillation vial.
11.6.12 Add 10 mL of Ultima Gold LLT or equivalent cocktail and shake thoroughly.
11.6.13 Inspect each LSV to ensure that it is free of dirt, grease, ect. Using an anti static "dryer" sheet, wipe the sides of the LSV to reduce static.

11.6.14 Submit samples to count room for analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 There are no calculations pertaining to this sample preparation procedure.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTEMANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA Method 906.0 "Tritium in Drinking Water"

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual Method, H3-04-RC

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM)

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (ST-HS-0002).

17.5 Associated SOPs

17.5.1 ST-RC-0002, "Preparation of Stainless Steel Planchets for Radiochemistry Analyses."
17.5.2 ST-RC-5006, "Decontamination of Laboratory Glassware. Labware and Equipment"
17.5.3 ST-RD-0302, "Operation and Calibration of a Liquid Scintillation Counter".
17.5.4 ST-QA-0002, "Standards and Reagent Preparation."
17.5.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.5.6 ST-QA-0036, "Non-conformance Memorandum (NCM) Process."
17.5.7 ST-PM-0002, "Sample Receipt and Chain of Custoday."

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 EPA Method 906.0 has been expanded to include soil samples.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Rev. 10:

19.1.1 Revised equipment and supplies needed in section 6.0.
19.1.2 Updated Standards and Reagents in section 7.0.
19.1.3 Updated Procedural methods in section 11.0.

19.2 Rev. 11:

19.2.1 No Changes, Annual Review.

19.3 Rev. 12:

19.3.1 Listed background water as dead water in section 7.0.
19.3.2 Replaced the use of boiling chips with sand throughout section 9.0.
19.3.3 Defined the cocktail for dead water in section 10.1.1.
19.3.4 Updated other kinds of solids used in section 11.3.

19.4 Rev. 13:

19.4.1 Updated the instructions for analyzing the method blank and the LCS in section 9.2 and 9.3.

19.5 Rev. 14:

19.5.1 Added the requirement for a post-distillation spike for silica gel treated samples in section 9.4.

19.6 Rev. 15:

19.6.1 Updated § 11.2.11 to clarify language on post distillation spike.

19.6.2 In section 7.0, updated instructions for verifiying cocktail mixtures used in analysis.

19.6.3 Updated instructions for verifiying sample pH before analysis in section 8.0.

19.6.4 Updated sections 9.2 and 9.3 to require rotation of condensors used for method blanks and LCS's.

19.7 Rev. 16:

19.7.1 Updated section 15.

19.7.2 Updated supplies in section 6.0.

19.7.3 Updated silica gel procedures and solids in section 11.2 and 11.3.

19.7.4 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.

19.7.5 Removed references to 'Clouseau" and "Quantims", replaced with LIMS.

19.8 Rev. 17:

19.8.1 Section 6.9, added 500-mL round and flat bottom flask to equipment and supplies used.

19.8.2 Grammatical errors fixed throughout

19.8.3 Section 8, Removed holding time

19.8.4 updated section 7.3 to detail process of using dead water to create background water

Title: RADIUM-226 AND RADIUM-228 BY CHEMICAL SEPARATION PREPARATION

Approvals (Signature/Date):

Sarah Bernsen 7/31/14 Sarah Bernsen Date Radiochemistry Prep Supervisor	7/31/14 Michael Ridenhower Date Health & Safety Manager / Coordinator
7/31/14 Marti Ward Date Quality Assurance Manager	Elaine Wild 7/31/14 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0041 Rev. 12

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1.0 SCOPE AND APPLICATION

1.1 This method covers the chemical separation preparation for radium-226 and radium-228 by direct measurement of its beta emitting progeny, Actinium (^{228}Ac). It is applicable to liquid (e.g water and wastewater) where complete dissolution and carrier exchange are readily achievable in the laboratory. For media where chemical dissolution is impractical, non-destructive measurement of the three principal photons of ^{228}Ac by gamma spectrometry is better suited.

1.2 This SOP is based on EPA Method 904.0, 903.0, SW9315, and SW9320.

1.3 The barium sulfate precipitate from this procedure contains all radium isotopes.

1.4 The barium sulfate can be counted for total alpha radiation. The time of the last barium sulfate precipitation should be recorded and used in calculating the in-growth factor.

1.5 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Radium isotopes are collected by coprecipitation with barium and lead sulfate and purified by precipitation from EDTA solution. After a suitable ingrowth period, ^{228}Ac is separated and carried on yttrium oxalate, purified and counted for the presence of total beta radiation. The precipitation and counting are performed in a manner consistent with the time requirements of the 6.13 hour half life of ^{228}Ac. By applying correction factors for ingrowth and decay and appropriately calibrating the beta counter, radium-228 is quantified. The barium sulfate fraction, minus radium-224, is counted by Gas Flow Proportional Counting (GFPC) to report radium-226.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 Strontium 90 or other beta emitting radionuclides that are carried by the yttrium oxalate precipitate (i.e. certain mixed fission or activation products) will yield a positive bias to the radium-228 values.

4.2 Samples which contain natural barium cause inaccurate chemical yield determinations.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [(1)]	Hazards	Exposure Limit [(2)]	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Acetic Acid, Glacial	Corrosive Flammable	10 ppm (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-(TWA) 4 ppm-(STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M^3-(TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Centrifuge tubes, 50-mL

6.2 Centrifuge

6.3 Hot Plate

6.4 Analytical balance (four decimal places)

6.5 Stainless steel planchets

6.6 Beakers, various volumes

6.7 Syringe, 20-mL; .45 mm filter

6.8 Water Bath

6.9 Desiccator

6.10 Pipette

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI water from the Millipore unit.

7.3 Acetic acid, 17.4 N: glacial CH_3COOH (concentrated), specific gravity 1.05, 99.8%.

7.4 Ammonium hydroxide, 15 N: NH_4OH (concentrated), sp. gr. 0.90, 56.6%.

7.5 Ammonium oxalate, 5%: Dissolve 2 g $(NH_4)_2C_2O{\cdot}H_2O$ in water and dilute to 40 mL.

7.6 Ammonium sulfate, 200 mg/mL: 400 g $(NH_4)_2SO_4$ in water and dilute to 2000 mL.

7.7 Ammonium sulfide, 2%: Dilute 5 mL $(NH_4)_2S$, (20-24%), to 50 mL water; total volume 50 mL

7.8 Barium carrier (standardized), 33.9 mg/mL, BaSO4 (20 mg/ml Ba)

7.8.1 If the barium carrier is not already standardized, standardize the barium carrier solution using the following procedure.

7.8.2 Pipette 1.0 mL barium carrier solution (20 mg/mL, Ba) into six separate labeled centrifuge tubes containing 15 mL DI H_2O.

7.8.3 To each tube, add 1 mL 18N sulfuric acid while stirring and digest precipitate in a hot water bath for approximately 10 min.

7.8.4 Cool, centrifuge and decant the supernate into appropriate waste container.

7.8.5 Wash precipitate with 15 mL DI water, centrifuge and decant the supernate.

7.8.6 Transfer the precipitate to a pre-weighed stainless steel planchet with a minimal amount of DI water.

7.8.7 Dry on a heat source. Store in desiccator until cool and weigh as barium sulfate.

7.8.7.1 Record the net weights of the precipitates and calculations in the Rad Standards Preparation Log.

7.9 Citric acid, 1M: Dissolve 192 g $C_6H_8O_7{\bullet}H_2O$ in water and dilute to 1000 mL.

7.10 EDTA reagent basic (0.25M) - dissolve 20 g NaOH in 750 mL water, heat and slowly add 93 g [ethylenedinitrilo] tetraacetic disodium salt, $(C_{10}H_{14}O_8N_2Na_2{\bullet}2H_2O)$ while stirring. Dilute to 1 L.

7.11 Lead carrier, 15 mg/mL: Dissolve 23.97 g $Pb(NO_3)_2$ in water, add 5 m: 16N HNO_3 and dilute to 1000 mL with water.

7.12 Lead carrier, 1.5 mg/mL: Dilute 10 mL lead carrier, (15 mg/mL), to 100 mL with water.

7.13 Methyl orange indicator, 0.1%: Dissolve 0.1g methyl orange indicator in 100 mL water.

7.14 Nitric acid, 16N: HNO_3 (concentrated), specific gravity 1.42, 70.4%.

7.14.1 Nitric acid, 6N: Mix 3 volumes 16N HNO_3 (concentrated) with 5 volumes of water.
7.14.1.1 Nitric acid, 2N: Mix 1 volume 6N HNO_3 with 2 volumes of water.

7.15 Sodium hydroxide, 18N: Dissolve 72g NaOH in water and dilute to 100 mL.

7.16 Sodium hydroxide, 10N: dissolve 40g NaOH in water and dilute to 100 mL.

7.17 Strontium carrier, 10 mg/mL: Dissolve 24.16 g $Sr(NO_3)_2$ in water and dilute to 1 liter.

7.18 Sulfuric acid, 18N: concentrated.

7.19 Yttrium carrier(standardized), 5 mg/ml

7.19.1 If the yttrium carrier is not already standardized, standardize the yttrium carrier solution using the following procedure.

7.19.1.1 Prepare 10 samples of the carrier solution. Perform the following steps on each vial.
7.19.1.2 Add 5 mL DI water to a 50 mL centrifuge tube.
7.19.1.3 Add 2 mL concentrated nitric acid.
7.19.1.4 Add 1 drop of MCP indicator (0.1%).
7.19.1.5 Add 1.0 mL of yttrium carrier (10 mg/mL, Y) pipette.
7.19.1.6 Add 5 mL of 0.4 M oxalic acid.
CAREFULLY add concentrated ammonium hydroxide dropwise until a reddish yellow end point is obtained.
NOTE: The pH must be between 1.7 - 1.9 to assure a uniform $9 \cdot H_2O$ hydrate yttrium oxalate precipitate. The yttrium oxalate will have precipitated.
7.19.1.7 Heat the solution for approximately 5 –10 minutes in a hot water bath.
7.19.1.8 Cool the solution for approximately 5 – 10 minutes in an ice water bath.
7.19.1.9 Centrifuge.
7.19.1.10 Decant supernate into the appropriate waste container.
7.19.1.11 Add approximately 20 mL of DI water to the precipitate.
7.19.1.12 Vortex and centrifuge.
7.19.1.13 Decant supernate into the appropriate waste container.
7.19.1.14 Add approximately 20 mL of DI water to the precipitate.
7.19.1.15 Vortex and Centrifuge.
7.19.1.16 Decant supernate into the appropriate waste container.
7.19.1.17 Add approximately 5 mL of D.I. water, slurry the precipitate.
7.19.1.18 Pre-weigh a planchet and record the tare weight on the sample worksheet.
7.19.1.19 Transfer the slurry to the planchet.
7.19.1.20 Dry the planchet on a hotplate.
7.19.1.21 Remove from heat and cool to room temperature in a desiccator.
7.19.1.22 Weigh sample to determine yttrium yield.
7.19.1.23 Repeat heating and cooling in the desiccator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are ± 5% or less.
7.19.1.24 Record gross and final weights in the Rad Standards Log.

7.20 Yttrium carrier, 2.5 mg/mL: Dilute 50 mL yttrium carrier, to 100 mL with water.

7.21 Strontium-yttrium mixed carrier, 0.9 mg/mL Sr^{+2}; 0.9 mg/mL Y^{+3}:
- 7.21.1 Solution A: Dilute 10.0 mL yttrium carrier, (18 mg/mL), to 100 mL
- 7.21.2 Solution B: Dissolve 0.4348 g $Sr(NO_3)_2$ in water and dilute to 100 mL.
- 7.21.3 The mixed carrier is made by adding equal volumes to a flask.

7.22 Radium-226, standard 20-25 dpm

7.23 Radium-228, standard 20-25 dpm

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 **Batch**
- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch comprises of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a <u>method blank</u>, a <u>Laboratory Control Sample</u> (LCS), and <u>Sample Duplicate</u>. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
  - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.
- 9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**
- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For Liquid analyses, the method blank is comprised of DI water acidified with 2 mL of nitric acid.
- 9.2.4 For Soil analyses, the method blank is comprised of DI water acidified with 2 mL of nitric acid.

9.3 **Laboratory Control Sample**
- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.
9.3.3 For Liquid analyses, the LCS is comprised of DI water fortified with radium-226 and radium-228.
9.3.4 For Soil analyses, the LCS is comprised of radium-226 and radium-228.

9.4 **Matrix Spike/Matrix Spike Duplicate**
9.4.1 A Matrix Spike (MS) is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch. MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate**
9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of an LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**
9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and thermometer calibration must be checked daily when used. Refer to SOP ST-QA-0005.

10.2 See the analytical SOP for instrument calibration; ST-RD-0403

11.0 PROCEDURE

11.1 Water Samples
11.1.1 Ensure that the sample container is capped tightly and shake it thoroughly. Ensure that the sample is at or below a pH of 2. Transfer a sample aliquot to a beaker.
11.1.2 Sample aliquot size is 1 liter.
11.1.2.1 For client requesting a reporting limit less than 1pCi/L, a larger sample volume may be required. Contact Radiochemistry manager/supervisor for instruction.
11.1.2.2 If less than 1 liter of sample was provided by the client, write the NCM noting insufficient volume.

11.2 Soil Samples
11.2.1 For soil samples prepare per SOP ST-RC-0003, “Drying and Grinding of Soil and Solid Samples”, and weigh 1 to 2 grams into a labeled crucible.
11.2.2 Place in oven at 600 °C and allow to muffle for four hours. Allow to cool.
11.2.3 Transfer to digestion tube using 4M HNO_3.

11.2.4 Add 1 mL of standardized barium carrier to samples and QC. Add radium-226 and radium-228 spike to LCS and MS/MSD, if applicable.
11.2.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
11.2.6 Digest in mod block at >110° for four hours or until dry.
11.2.7 Carefully add 10 mL concentrated nitric acid, 10 mL concentrated hydrochloric acid and 5 mL concentrated Hydrofluoric acid.
11.2.8 Digest in mod block at >110° for four hours or until dry.
11.2.9 Dissolve with 10 mL HNO_3 and 10 mL HCl, return to mod block for 30min.
11.2.10 Transfer to 250 mL beakers with 4M HNO_3. Dilute to 200 mL with DI water.

11.3 Precipitation
11.3.1 Add 1M citric acid in ratio of 5 mL per liter. Mix thoroughly. Add methyl orange indicator until red color persists.
11.3.2 For Waters perform steps 11.3.2.1, 11.3.2.2 and 11.3.2.3; for soils perform step 11.3.2.1
11.3.2.1 Add 2.5 mL lead carrier (15 mg/mL), 2 mL strontium carrier (10 mg/mL), and 1 mL yttrium carrier (9 mg/mL); stir well.
11.3.2.2 Add 1.0 mL standardized barium carrier (33.9 mg/mL),
11.3.2.3 Spike LCS and MS/MSD (if applicable) with Radium 226 and radium-228.
11.3.3 Heat to just boiling and maintain at this temperature for about 5-10 minutes.
11.3.4 Add 15N ammonium hydroxide until a definite yellow color is obtained, then add a few drops more.
11.3.5 **Face shield must be worn during this process.** Precipitate lead and barium sulfates by adding 10 mL of 18N sulfuric acid, or until the red color reappears.
11.3.6 Add 5 mL ammonium sulfate (200 mg/mL) for each liter of sample. Stir frequently and keep at a temperature of approximately 90°C for 30 minutes.
11.3.7 Allow precipitate to settle to the bottom of the beaker for a least 6 hours.

11.4 In-growth
11.4.1 Decant the supernate and discard, taking care to avoid disturbing the precipitate. Transfer precipitate to a 50 mL centrifuge tube, taking care to rinse last particles out of beaker with DI water. Centrifuge and discard supernate.
11.4.2 Wash the precipitate with 10 mL 16N HNO_3, vortex, centrifuge, and discard supernate. Repeat this step if precipitate visually appears larger than that in the Blank and LCS.
11.4.3 Wash the precipitate with 10 mL D.I.H_2O, vortex, centrifuge, and discard supernate.
11.4.4 Add 1 mL strontium-yttrium mixed carrier.
11.4.5 Add 20 mL basic EDTA reagent; vortex thoroughly, and heat in a hot water bath (approximately 80°C) until precipitate dissolves. There should be no precipitate remaining in the tube.
11.4.5.1 If insoluble solids remain in the tube after addition of EDTA, confirm that the pH is > 10.
11.4.5.2 If pH > 10, centrifuge and syringe filter the supernate into a clean, labeled 50 mL centrifuge tube. Discard insoluble residue.
11.4.6 Add 2 mL ammonium sulfate (200mg/mL) and stir thoroughly.
11.4.7 Add 3 mL of 17.4N acetic acid or until barium sulfate precipitates.
11.4.8 Digest in a hot water bath until precipitate settles.
11.4.9 Centrifuge and discard supernate.
11.4.10 Add 20 mL basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.
11.4.11 Add 2 mL ammonium sulfate (200mg/mL) and stir thoroughly.
11.4.12 Add 3 mL of 17.4N acetic acid or until barium sulfate precipitates
11.4.13 Record the date and time of last barium sulfate precipitation. This is the beginning of the ^{228}Ac in-growth time.
11.4.14 Digest in a hot water bath until precipitate settles.

11.4.15 Centrifuge and discard supernate.
11.4.16 Add 20 mL basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.
11.4.17 Add 1.5 mL standardized yttrium carrier and 1 mL lead carrier (1.5 mg/mL).
11.4.17.1 If any precipitate forms, dissolve it by adding a few drops of 10N NaOH.
11.4.17.2 If radium-228 analysis is requested, cap the tube and allow it to age at least 36 hours.
11.4.17.3 If radium-226 analysis is requested, cap the tube and allow it to age (in-growth period is 14 or 21 days, see sample log in sheet).

11.5 Lead Scavenge Clean-up
11.5.1 Add 0.3 mL ammonium sulfide. Add 0.5 mL of 10N sodium hydroxide until lead sulfide precipitates, vortex than centrifuge.
11.5.2 Add 1 mL lead carrier (1.5 mg/mL), 0.1 mL ammonium sulfide, and 0.1 mL of 10N sodium hydroxide.
11.5.3 Centrifuge and filter supernate through 0.45 mm syringe filter into a clean labeled tube. Discard filter.
11.5.3.1 The half life ^{228}Ac is very short. Check with count room staff on scheduling of the GFPC. **Do NOT proceed with the remaining steps of this procedure until authorized by the count room GFPC analysts or Radiochemistry manager.**

11.6 Out of In-growth
11.6.1 Once yttrium hydroxide is precipitated, the analysis must be carried to completion to avoid excessive decay of ^{228}Ac.
11.6.2 Add 10 mL 18N sodium hydroxide, stir well and digest in a hot water bath, 70-85°C, until yttrium hydroxide coagulates, usually about 5 minutes. Centrifuge and decant supernate into a clean, labeled 50 mL centrifuge tube. Save for barium yield determination, Step 11.7.1

11.7 Yttrium Yield (for radium-228)
11.7.1 Weigh a stainless steel cleaned planchet. Record weight in the LIMS spreadsheet.
11.6.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.
11.7.2 Record time of yttrium hydroxide precipitation; this is the end of the ^{228}Ac in-growth time and beginning of ^{228}Ac decay time.
11.7.3 Dissolve the precipitate from step 11.5.2 in 2 mL 6N nitric acid. Vortex and add 5 mL water and precipitate yttrium hydroxide with 3 mL 10N sodium hydroxide. Heat and stir in a hot water bath until precipitate coagulates. Centrifuge and discard supernate.
11.7.4 Dissolve precipitate with 1 mL 2N nitric acid. Vortex.
11.7.4.1 If solution is still cloudy add 2N nitric acid drop wise until the solution clears.
11.7.5 Dilute to 5 mL with DI water and add 2 mL 5% ammonium oxalate. Centrifuge and discard supernate.
11.7.6 Wash precipitate with 10 mL DI water.
11.7.7 Vortex, centrifuge and discard supernate.
11.7.8 Plating
11.7.8.1 To determine yttrium yield, quantitatively transfer the precipitate to the previously weighed stainless steel cleaned planchet using a minimal amount of water.
11.7.8.2 Dry on a hot plate.
11.7.8.3 Upon dryness, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.
11.7.8.4 Record the weight in the LIMS worksheet to determine the chemical yield of the yttrium carrier solution.

11.8 Barium Yield (for radium-226)

11.8.1 Weigh a stainless steel cleaned planchet. Record weight in the LIMS spreadsheet.

11.8.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.

11.8.2 To the supernate from Step 11.5.2, add 7 mL 16N nitric acid and 2 mL ammonium sulfate (200mg/mL),. Add 7 mL 17.4N acetic acid until barium sulfate precipitates. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.

11.8.3 Add 20 mL basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitates does not readily dissolve.

11.8.4 Add 2 mL ammonium sulfate (200 mg/mL) and stir thoroughly. Add 3 mL 17.4N acetic acid until barium sulfate precipitates

11.8.5 Record date and time of $BaSO_4$ precipitate in the LIMS data sheet.

11.8.6 Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.

11.8.7 Wash precipitate with 10 mL water. Vortex, centrifuge and discard supernate.

11.8.8 Repeat wash step.

11.8.9 Plating

11.8.9.1 Transfer the precipitate to a pre-weighed stainless steel cleaned planchet with a minimal amount of water.

11.8.9.2 Heat the planchet again using the hot plate, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.

11.8.9.3 Record the final weight of the planchet in LIMS to determine the chemical recovery for the barium carrier solution.

11.9 Submit the planchets to the counting room for analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. LCS % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 There are no calculations pertaining to this sample preparation procedure.

12.3 Radium-226 and radium-228 by GFPC calculations are given in ST-QAM.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type “A” or “B”.

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid Rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0, Radium-228 in Drinking Water

17.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0, Alpha-Emitting Radium Isotopes in Drinking Water

17.3 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods”, Method 9315, Alpha Emitting Radium Isotopes

17.4 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods”, Method 9320, Radium-228

17.5 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.6 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revision

17.7 Associated SOPs:

17.7.1 ST-PM-0002, Sample receipt and Chain of Custody
17.7.2 ST-QA-0002, Standard and Reagent Preparation
17.7.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.7.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.7.5 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis
17.7.6 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.7.7 ST-RD-0403, Gas Flow Proportional Counting (GFPC) Analysis

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 After initial precipitation, TestAmercica St. Louis decants the supernate after the precipitate has been allowed to settle for at least six hours, as opposed to the EPA Method 904 which requires filtration to isolate the precipitate.

18.2 At the point of in-growth of actinium-228, TestAmerica St. Louis utilizes either a 14 days or 21 day in-growth depending on the client's request for wastewater and 21 days for drinking water before finishing the procedure.

18.3 TestAmerica St. Louis counts the barium sulfate fraction (minus the Ra-224) by GFPC to report radium-226; an option documented in section 10.5 of the EPA Method 903.0.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Annual Review, No Changes.
19.2 Rev. 10:
- 19.2.1 Updated section 11.4 to add carrier prior to adding EDTA.
- 19.2.2 Moved 11.4.15, recording the date and time of the last barium sulfide precipitation to section 11.4.9.

19.3 Rev. 11:
- 19.3.1 Updated soil LCS composition in section 9.3.4.
- 19.3.2 Updated amount of standardized yttrium carrier used in section 11.4.17
- 19.3.3 Updated amount of DI water for precipitate wash and removed repetition of wash and vortex steps in section 11.7.7 and 11.7.8.

19.4 Rev. 12:
- 19.4.1 Grammatical corrections throughout
- 19.4.2 RadCap/RadCapture changed to LIMS.
- 19.4.3 Deleted stir thoroughly
- 19.4.4 Section 11.3.3 changed time from 30 minutes to 5-10 minutes.
- 19.4.5 Section 11.3.5 added: add 10 mL, and deleted add 0.25 mL more.
- 19.4.6 Section 11.4.5.2 added syringe filter supernate into clean labeled tube.
- 19.4.7 Section 11.4.7 changed aliquot to 3 mL of acetic acid.
- 19.4.8 Section 11.4.9 moved to after 11.4.13.
- 19.4.9 Section 11.4.13 changed aliquot of acetic acid to 3 mL.
- 19.4.10 Section 11.5.1 removed stir well after ammonium sulfide is added, and changed aliquot of sodium hydroxide to 0.5 mL.
- 19.4.11 Section 11.5.2 changed aliquot of sodium hydroxide to 0.1 mL.
- 19.4.12 Section 11.8.2 deleted stir well after each addition and changed aliquot to 7 mL acetic acid.
- 19.4.13 Section 11.8.4 changed aliquot of acetic acid to 3 mL.
- 19.4.14 Updated section 15

19.5 Revision 13:
- 19.5.1 Section 7.8 added the name of the reagent, BaSO4

19.5.2 Section 7.8.2 changed 16 to 20 mg/ml, Ba per certificate
19.5.3 Section 7.17 made correction fo the concentration , replaced 9 with 10 mg/ml per method.
19.5.4 Section 7.19 added concentration, 5 mg/ml
19.5.5 Section 7.2 Corrected concentration from 9 to 2.5 mg/ml.

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Title: AQUEOUS AND SOIL SAMPLE PREPARTION FOR STRONTIUM-89, STRONTIUM-90 AND TOTAL STRONTIUM

Approvals (Signature/Date):

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the process for sample preparation of Strontium-89, Strontium-90 and Total Strontium. This procedure is applicable to aqueous samples and may be used in conjunction with soil pre-preparation procedures to perform Strontium determinations in solid matrices.

1.2 This SOP is based on EPA Method 905 and DOE EML HASL 300 Method, Sr-03-RC, Sr-02..

1.3 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Strontium is precipitated from a liquid matrix sample as insoluble carbonate. For soils, the Strontium is transferred from the soil into a liquid matrix prior to precipitation. Interferences from calcium and other radionuclides are removed by one or more precipitations of the Strontium carrier as Strontium nitrate. Samples with high dissolved solids can be precipitated as sulfates from an acid solution, eliminating most of the calcium interference. DTPA is added to the samples precipitated as sulfates to further remove interferences. Barium and radium are removed as chromate. The yttrium-90 daughter of Strontium-90 is removed by a hydroxide precipitation step. The yttrium-90 daughter grows in again and is then separated with stable yttrium carrier as hydroxide and finally precipitated as oxalate and beta counted. Strontium-90 and Strontium Total is counted for beta particle activity by Gas Flow Proportional Counters (GFPC). The Strontium-89 concentration is determined by difference.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 Samples which contain natural strontium cause inaccurate chemical yield determinations

4.2 In hard water, large quantities of calcium precipitate with the Strontium in the initial carbonate precipitation. Repeated washes with concentrated nitric acid and precipitation of calcium as sulfate minimizes this potential interference.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid, Glacial	Corrosive Flammable	10 ppm (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Ammonium Hydroxide	Poison Corrosive	50 ppm - (TWA) (NH_3)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Fuming Nitric Acid	Poison Corrosive Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation symptoms include breathing difficulties, coughing, choking, and irritation of the nose, throat, and respiratory tract. Onset of symptoms may be delayed 4-30 hours. Ingestion of nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract. Skin contact can cause redness, pain, and severe skin burns. Vapors are irritating to the eyes.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
Oxalic Acid	Corrosive	1mg/m^3 (TWA) 2mg/m^3 (STEL)	Inhalation symptoms include severe irritation and burns of nose, throat, and respiratory tract. Ingestion symptoms include burns, nausea, severe gastroenteritis and vomiting. Skin contact causes severe irritation and burns. Oxalic Acid is an eye irritant.
Sodium Chromate	Corrosive Oxidizer Cancer Causing	For chromic acid and chromates, as CrO_3 = 0.1 mg/m^3 (ceiling)	Inhalation symptoms may include sore throat, coughing, shortness of breath, and labored breathing. Skin contact may include redness, pain, and severe burning. Eye contact can cause blurred vision, redness, pain and severe tissue burns.
Sodium Hydroxide	Corrosive Poison	2 mg/m^3 (TWA)	Nose irritation; pneumonitis; eye, skin burns; temporary loss of hair
Hydrochloric Acid	Poison Corrosive	5 ppm (ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Hydrofluoric Acid	Poison Corrosive	3 ppm (TWA)	Inhalation symptoms may include sore throat, coughing, labored breathing and lung congestion/inflammation. Skin contact may cause serious burns which are not immediately apparent or painful. Symptoms of eye contact include redness, pain, and blurred vision.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical Balance (min. 4 place)

6.2 Centrifuge and centrifuge tubes

6.3 Quartz crucibles

6.4 Glass and teflon beakers, various sizes, covers and watch glasses

6.5 Planchets

6.6 Spatulas

6.7 Hot Plate

6.8 Hot bath

6.9 pH paper, narrow range

6.10 Desiccator

6.11 Digestion Vessels

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Deionized Water (DI water), obtained from the Milli-Q unit.

7.3 Acetic Acid (17.4 M), glacial CH_3COOH.

7.4 Ammonium Hydroxide (15 M) - concentrated NH_4OH.

7.5 Barium carrier

7.5.1 Barium carrier (10 mg/mL, Ba) - In a 1-L graduated cylinder, dissolve 19 g of Barium Nitrate in 500 mL of deionized water. With stirring carefully, add 5 mL of concentrated Nitric Acid and dilute to a final volume of 1 L with deionized water.

7.6 DTPA 0.2 M, Diethylenetriaminepentaacetic Acid, Pentasodium Salt (40 % in H_2O)., Dilute 200 mL of DTPA solution to 1000 mL with DI water.

7.7 Hydrochloric acid (12 M HCl) - concentrated, 37.2%.

7.8 Metacresol Purple Indicator (MCP) (0.1%) - In a volumetric flask, dissolve 0.5g of m-cresolsulfonephthalein (MCP) in deionized water. Add 2 mL of 10M NaOH and dilute to a final volume of 500 mL with deionized water.

7.9 Nitric Acid (90%) – fuming.

7.10 Nitric Acid (16 N) (69 – 71%) - concentrated HNO_3.

7.11 Hydrofluoric Acid, concentrated (29 N HF)

7.12 Oxalic Acid (0.4 M) - In a 1-L graduated cylinder, dissolve 52.02 g of oxalic acid, ($H_2C_2O_4$), in deionized water. Dilute to a final volume of 1 L with deionized water. Mix thoroughly.

7.13 Sodium Carbonate ($Na_2CO_3 \cdot 10H_2O$), (1 M) - In a 1-L graduated cylinder, dissolve 286 g of sodium carbonate, in deionized water. Dilute to a final volume of 1 L with deionized water. Mix thoroughly.

7.14 Sodium Chromate (Na_2Cr0_4), (1.5 M) - In a 1-L beaker, dissolve 242.96 g of sodium chromate in deionized water. Add the solution to a graduated cylinder. Dilute to a final volume of 1 L with deionized water. Mix thoroughly.

7.15 Sodium Hydroxide (NaOH), (10 M) - In a 1 L beaker, SLOWLY and CAREFULLY dissolve 400 g of sodium hydroxide pellets, in approximately 500 mL of deionized water. When the solution has cooled to room temperature, quantitatively transfer the solution to a graduated cylinder. Dilute to a final volume of 1 L with deionized water.

7.16 Sodium Sulfate, Na_2SO_4, crystals.

7.16.1 Sodium Sulfate (Na_2SO_4), (0.7 M) - In a 1 L graduated cylinder, dissolve 100 g of sodium sulfate in deionized water. Dilute to a final volume of 1 L with deionized water.

7.17 Strontium carrier

7.17.1 Strontium Solution (10 mg/mL, Sr) In a 1-L volumetric flask, dissolve 12.077 g of Strontium Nitrate in approximately 250 mL of deionized water. With stirring, add 5 mL concentrated Nitric Acid. Dilute to a final volume of 1 L with deionized water.

7.18 Strontium Carrier (standardized) 52.5 mg/mL

7.18.1 If the Strontium carrier is not already standardized, standardize the Strontium carrier using the following procedure:

7.18.1.1 Prepare 3 samples of the carrier solution. Perform the following steps on each vial.
7.18.1.2 Add 6 mL of 0.2M DTPA to a 50-mL centrifuge tube.
7.18.1.3 Add 1.00 mL Strontium carrier
7.18.1.4 Add 2 drops metacresol purple indicator.
7.18.1.5 Add 10M NaOH with swirling until a purple endpoint is achieved.
7.18.1.6 Add 10 mL of 0.7M sodium sulfate.
7.18.1.7 Add approximately 3 mL of glacial acetic acid with swirling until a yellow solution is achieved.
7.18.1.8 Heat the solution for 5 - 10 minutes in a hot water bath.
7.18.1.9 Cool for 5 - 10 minutes in an ice bath.
7.18.1.10 Centrifuge for approximately 10 minutes at a speed sufficient to cause the precipitate to form a pellet.
7.18.1.11 Decant and discard the supernate in the appropriate waste container.
7.18.1.12 To the precipitate, add 20 mL of DI water
7.18.1.13 Vortex well, Centrifuge and decant.
7.18.1.14 Add 20 mL of DI water.
7.18.1.15 Vortex well, Centrifuge and decant.
7.18.1.16 Add approximately 5 mL of deionized water, slurry the precipitate.
7.18.1.17 Pre-weigh a planchette and record the tare weight.
7.18.1.18 Transfer onto a preweighted planchette with a spatula.
7.18.1.19 Heat to dryness on a hotplate at medium heat.
7.18.1.20 Remove from heat and cool to room temperature in a dessicator.
7.18.1.21 Weigh sample to determine Strontium yield.

7.18.1.22 Repeat heating and cooling in the dessicator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are ± 5% or less.

7.18.1.23 Record gross and final weights in the Rad Standards Log. Assign a unique number to the solution.

7.19 Ytrium carrier (standardized)

7.19.1 If the Yttrium carrier is not already standardized, standardize the Yttrium carrier using the following procedure:

7.19.1.1 Prepare 3 samples of the carrier solution. Perform the following steps on each vial.

7.19.1.2 Add 5 mL deionized water to a 50 mL centrifuge tube.

7.19.1.3 Add 2 mL concentrated nitric acid.

7.19.1.4 Add 1 drop of MCP indicator (0.1%).

7.19.1.5 Add 1.0 mL of yttrium carrier (10 mg/mL, Y)

7.19.1.6 Add 5 mL of 0.4 M oxalic acid.

7.19.1.7 **CAREFULLY add concentrated ammonium hydroxide dropwise until a reddish yellow end point is obtained.**
NOTE: The pH must be between 1.7 - 1.9 to assure a uniform 9·H_2O hydrate yttrium oxalate precipitate. The yttrium oxalate will have precipitated.
Heat the solution for approximately 5 –10 minutes in a hot water bath.

7.19.1.8 Cool the solution for approximately 5 – 10 minutes in an ice water bath.

7.19.1.9 Centrifuge.

7.19.1.10 Decant supernate into the appropriate waste container.

7.19.1.11 Add approximately 20 mL of DI water to the precipitate.

7.19.1.12 Vortex. and Centrifuge.

7.19.1.13 Decant supernate into the appropriate waste container.

7.19.1.14 Add approximately 20 mL of DI water to the precipitate.

7.19.1.15 Vortex and Centrifuge.

7.19.1.16 Decant supernate into the appropriate waste container .

7.19.1.17 Add approximately 5 mL of D.I. water, slurry the precipitate.

7.19.1.18 Pre-weigh a planchet and record the tare weight on the sample worksheet.

7.19.1.19 Transfer the slurry to the planchette.

7.19.1.20 Dry the planchette on a hotplate.

7.19.1.21 Remove from heat and cool to room temperature in a desiccator.

7.19.1.22 Weigh sample to determine yttrium yield.

7.19.1.23 Repeat heating and cooling in the dessciator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are ± 5% or less.

7.19.1.24 Record gross and final weights in the Rad Standards Log. Assign a unique number to the solution.

7.20 Strontium-90

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 The pH of aqueous samples are checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis.

8.3.1 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior ro analysis.

8.4 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.5 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Liquid analyses, the method blank is comprised of 1 L DI water acidified with 2ml nitric acid.

9.2.4 For Soil analyses, the method blank is comprised of DI water acidified with 2ml nitric acid.

9.2.5 For leached analyses, the method blank iscomprised of the leaching acid.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Liquid analyses, the LCS is comprised of 1 L DI water acidified with 2ml nitric acid fortified with Strontium-90.

9.3.3.1 For Strontium-90 and/or Total Strontium, the LCS is fortified with Strontium 90.

9.3.4 For Soil analyses, the LCS is comprised of with Strontium-90.

9.3.4.1 For Strontium-90 and/or Total Strontium, the LCS is fortified with Strontium-90.

9.3.5 For leached analyses, the LCS comprised of the leaching acid fortified with Strontium-90.

9.4 **Matrix Spike/Matrix Spike Duplicate**

9.4.1 A Matrix Spike (MS) is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of an LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and automatic pipetter calibrations must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

10.2 See analytical SOP ST-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System", for calibration requirements.

11.0 PROCEDURE

11.1 Water Samples

11.1.1 Initiate sample preparation worksheet.

11.1.2 Ensure that the sample container is capped tightly and shake it thoroughly.

11.1.3 Transfer a 1 L sample aliquot to a beaker.

11.1.3.1 For client requesting a reporting limit less than 2 pCi/L, a larger sample volume may be required. Contact Radiochemistry manager/supervisor for instruction.

11.1.3.2 If less than 1 L of sample was provided by the client, write the NCM noting insufficient volume.

11.1.3.3 Upon visual inspection, if the aqueous samples is suspected to have a high density (> 1.2g/mL, e.g. a brine or waste) or a low density (> 0.98g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).

11.1.4 Add 2.0 mL standardized Strontium carrier to samples and QC. Add spike to LCS and MS/MSD, if applicable.

11.1.4.1 For Strontium-90 and/or Total Strontium, the LCS is fortified with Strontium-90.

11.1.5 Proceed to Preciptation Step 11.3.

11.2 Soil Samples

11.2.1 For soil samples prepare sample per SOP ST-RC-0003, "Drying and Grinding of Soil and Solid Samples."

11.2.2 Weigh 1 to 2 g into a labeled crucible.

11.2.3 Place in oven at 600° and allow to muffle for four hours. Allow to cool.

11.2.4 Transfer to digestion tube. Rinse using 4M HNO_3.

11.2.5 Add 2 mL of standardized Strontium carrier to samples and QC. Add spike to LCS and MS/MSD, if applicable.

11.2.5.1 For Strontium-90 and/or Total Strontium, the LCS is fortified with Strontium-90.

11.2.6 Carefully add 5 mL concnetrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.

11.2.7 Digest in mod block at >110° for four hours or until dry.

11.2.8 Carefully add 10 mL concnetrated nitric acid, 10 mL concentrated hydrochloric acid and 5 mL concentrated Hydrofluoric acid.

11.2.9 Digest in mod block at >110° for four hours or until dry.

11.2.10 Dissolve with 10 mL HNO_3 and 10 mL HCl, return to mod block for 30min.

11.2.11 Transfer to 400 mL beakers, rinsing with 4M HNO_3.

11.2.12 Bring up to volume of 200 mL with DI H_2O.

11.3 Precipitation

11.3.1 Add enough MCP indicator, while stirring, to see the red point of the indicator.

11.3.2 Add 1 mL of barium carrier (10 mg/mL).

11.3.3 Add 10 M sodium hydroxide (NaOH) to the purple point of the indicator.

11.3.4 Heat to near boiling as determined by visual observation.

11.3.5 SLOWLY, add 10 mL 1M Na_2CO_3 solution.

11.3.6 Heat the sample approximately 15 minutes, stirring occasionally. Turn off the heat. **NOTE: Sample(s) should become cloudy indicating the formation of a precipitate. If no precipitate is visible, continue to add sodium carbonate solution until precipitation is achieved. If no precipitate forms after 20 mL Na_2CO_3, inform manager/supervisor before continuing.**

11.3.7 Cover the sample and allow the precipitate to settle for at least 6 hours at room temperature.

11.4 Strontium and Barium Precipitation

11.4.1 Decant off the supernate and discard into the appropriate waste container

11.4.2 Transfer the precipitate to a labeled centrifuge tube, rinsing with DI water as needed to transfer.

11.4.3 Centrifuge for approximately 5 minutes at a speed sufficient to cause the precipitate to form the shape of a pellet.

11.4.4 Discard the supernate into the appropriate waste container.

11.4.5 Repeat the centrifuge steps (11.4.3 – 11.4.4) as necessary to collect the entire sample in a single centrifuge tube.

11.4.6 Slowly add 10 mL concentrated nitric acid to the precipitate in the centrifuge tube.
CAUTION: Visually confirm that the chemical reaction is completed before capping the tube. Gas may form which may cause the sample to splash.

11.4.7 Cap the tube. Mix the solution with a Vortex mixer until dissolved.
CAUTION: The next step must be performed in a fume hood that has been designated for strong oxidizing acids. Wearing a facesheild, CAREFULLY add the fuming nitric acid.

11.4.8 CAREFULLY and slowly add 20 mL fuming nitric acid to the centrifuge tube.

11.4.9 Cap centrifuge tube and cool in an ice bath for 10 minutes with occasional swirling to precipitate the Strontium and Barium Nitrates.

11.4.9.1 If a precipitate does not form, add additional fuming nitric acid in 10 mL increments until precipitate forms. Chill in an ice bath.

11.4.9.2 If after adding 20 mL of fuming nitric acid addition, a precipitate still has not formed, contact manager/supervisor for instruction.

11.4.10 Centrifuge for 5-10 minutes at a speed sufficient to cause the precipitate to form the shape of a pellet.

11.4.11 Discard supernate to acid waste container.

11.4.12 Add 10 mL concentrated nitric acid, vortex vigorously to completely break up the precipitate.

11.4.13 Heat in hot water bath for approximately 5 minutes.

11.4.14 Centrifuge for 5-10 minutes at a speed sufficient to cause the precipitate to form the shape of a pellet.

11.4.15 Discard supernate in the acid waste container.

11.4.16 Repeat steps 11.4.12to 11.4.15 until the mass of the sample appears similar to the mass of the method blank and LCS.

11.4.17 Dissolve the precipitate in 10 mL of deionized water.

11.4.18 Vortex**.**

11.4.18.1 If any insoluable solid remains, centrifuge for 5-10 minutes at a speed sufficient to shape any insoluble solids in the form of a pellet.

11.4.18.2 Transfer supernate to a unused labeled centrifuge tube. Set aside any insoluble residue.

11.5 Strontium Sulfate Precipitation

11.5.1 Add 2 drops MCP indicator.

11.5.2 Add 5 mL DTPA.

11.5.3 Dropwise, add 10 M sodium hydroxide to the sample with swirling to a purple color change.

11.5.3.1 Dropwise add 10 M sodium hydroxide in excess, color will not change.

11.5.4 Add 5 mL of 1.5 M sodium chromate.

NOTE: It is important to add the glacial acetic acid of the next step slowly so that it does not prematurely precipitate the Strontium.

11.5.5 While mixing CAREFULLY and SLOWLY, add 17.4 M glacial acetic acid dropwise until the color of the solution changes from golden yellow to reddish-orange. The pH should optimally be 5.3. Check pH with narrow range pH paper.

11.5.6 Heat for at least 5 minutes in a hot water bath.

11.5.7 Cool the solution for 5 - 10 minutes in an ice bath.

11.5.7.1 If a precipitate does not form, the solution may be “seeded” with 1-2 drops of Na_2CrO_4 mixed with 1-2 drops of Barium solution.

11.5.8 Centrifuge for approximately 5 minutes at a speed sufficient to form the precipitate into the shape of a pellet.

11.5.9 Transfer the supernate to a clean 50 mL unused, labeled centrifuge tube.

11.5.10 To the supernate add 10 mL of 0.7M sodium sulfate.

11.5.11 Add 7 mL 17.4M glacial acetic acid to precipitate the Strontium sulfate.

11.5.12 Centrifuge. Decant the supernate to the appropriate waste container.

11.5.13 Add approximately 10ml of DI water, vortex to slurry the solution. Centrifuge. Decant off the supernate the appropriate waste container.

11.5.13.1 If the precipitate is still yellow/orange, repeat DI water step, 11.5.13.

11.5.14 Record the current date and Time as "T1", the start date and time of Y-90 ingrowth, and TSR on the laboratory worksheet.

11.5.15 For Total Strontium (only) analyses proceed to 11.9.

11.6 Ingrowth

11.6.1 Dissolve the Strontium sulfate precipitate from Step 11.5.15 in 10 mL of 0.2 M DTPA.

11.6.2 Vortex thoroughly.

11.6.2.1 If precipitate is visually apparent, heat to complete dissolution.

11.6.3 Add 1.5 mL of Yttrium carrier

11.6.4 Add 1 drop MCP.

11.6.4.1 The color should be purple.
11.6.4.2 If the color is not purple, add 10 M NaOH, dropwise, until a purple color is obtained.
11.6.4.2.1 If more than 1 mL NaOH has been added and the color is not obtained, contact manager/supervisor for instruction.
11.6.5 Cap the tube and store for 7 to 14 days to permit ingrowth of Yttrium 90.

11.7 Out of In-growth
11.7.1 Add 5 mL of 0.7 M sodium sulfate.
11.7.2 Precipitate Strontium sulfate by adding 3 mL 17.4M glacial acetic acid.
11.7.3 Record the current date and time, "T2", as the end date and time of the in-growth period of ^{90}Y on the sample worksheet.
NOTE: From step 11.7.4 to step 11.10 are to be completed before the end of the working day. If all steps can not be completed within the prescribed time, do not start the next step. Contact manager/supervisor for instruction.
11.7.4 Heat the solution for 5 - 10 minutes in a hot water bath.
11.7.5 Cool the solution for 5 - 10 minutes in an ice bath.
11.7.6 Centrifuge for approximately 5 minutes at a speed sufficient to cause the precipitate to form a pellet.
NOTE: In the next section of this procedure, ^{89}Sr, ^{90}Sr is separated from ^{90}Y to determine a separate activity for ^{90}Y, and for Strontium.

11.8 Yttrium Yield
11.8.1 Transfer the supernate to a unused, labeled centrifuge tube.
11.8.1.1 **Cap and save the precipitate fraction for step 11.9**
11.8.2 To the supernate, add 2 drops of MCP indicator.
11.8.3 While swirling, add 15 mL 10M sodium hydroxide to the sample to achieve the purple endpoint.
11.8.4 Heat the solution for 5 - 10 minutes in a hot water bath.
11.8.5 Cool the solution for 5 - 10 minutes in an ice water bath.
11.8.6 Centrifuge for approximately 10 minutes at a speed sufficient to cause the precipitate to form a pellet.
11.8.7 Decant, discarding the supernate into the base waste container.
11.8.8 Dissolve the precipitate in 2 mL of concentrated nitric acid.
11.8.9 Add 5 mL of DI water.
11.8.10 Add 5 mL of 0.4 M oxalic acid.
11.8.11 Add 1 drop of MCP indicator.
11.8.12 CAREFULLY, add concentrated ammonium hydroxide dropwise until a pinky orange end point is obtained. **NOTE: The pH should be between 1.6 - 1.9 to assure a uniform 9·H_2O hydrate yttrium oxalate precipitate.** Verify pH with narrow range pH paper.
11.8.12.1 If pH > 1.9, contact manager/supervisor for instruction.

11.8.13 Heat the solution for approximately 5 minutes in a hot water bath.
11.8.14 Centrifuge for approximately 5 minutes. Decant supernate to waste.
11.8.15 Add approximately 10 mL of DI water to the sample.
11.8.16 Vortex to slurry the sample.
11.8.17 Centrifuge for approximately 5 minutes. Decant supernate to waste.
11.8.18 Plating
11.8.18.1 Pre-weigh planchet and record weight.
11.8.18.2 Add approximately 7 mL of DI water to the precipitate.
11.8.18.3 Slurry the precipitate.
11.8.18.4 Transfer sample slurry onto a pre-weighed planchette.
11.8.18.5 Dry the planchette on a hotplate.
11.8.18.6 Remove from heat and allow sample to cool to room temperature in a desiccator.

11.8.18.7 Upon dryness, let cool in a desiccator for a minimum of 20 minutes and then weigh planchet.

11.8.18.8 Record the weight in the RadCapture worksheet to determine the chemical yield of the yttrium carrier solution.

11.9 Strontium Yield

11.9.1 Add approximately 10 mL of DI water to the precipitate.

11.9.1.1 For Sr-89 and/or Sr-90, precipitate is from 11.8.1.1.

11.9.1.2 For Total Strontium, precipitate is from 11.5.15.

11.9.2 Vortex to slurry the sample.

11.9.3 Centrifuge for approximately 5 minutes. Decant supernate to waste.

11.9.4 Plating

11.9.4.1 Pre-weigh planchet and record weight.

11.9.4.2 Transfer the precipitate to a pre-weighed stainless steel cleaned planchet with a minimal amount of water.

11.9.4.3 Heat the planchet again using the hot plate, let cool in a desiccator for a minimum of 20 minutes and then weigh planchet.

11.9.4.4 Record the final weight of the planchet in RadCapture to determine the chemical recovery for the Strontium carrier solution.

11.10 Submit the planchet for counting to determine the Strontium activity.

11.10.1 If not able to submit to the count room, hold planchet in desiccator until ready.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Density of aqueous samples: sample mass divided by the density equals the volume.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1. Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS.

14.2. Demonstration of Capability

14.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3. Training Qualification

14.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Sr-03-RC, Strontium -90 in Environmental Samples, Sr-02, Strontium-90.

17.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water

17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs (Current Revisions)

17.5.1 ST-RC-0002, Preparation of Stainless Steel Planchets for Radiochemistry Analyses

17.5.2 ST-RC-0003, Drying and Grinding of Soil and solid Samples

17.5.3 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis

17.5.4 ST-RC-5006, Decontamination of Laboratory Glassware. Labware and Equipment

17.5.5 ST-RD-0403, Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System
17.5.6 ST-QA-0002, Standards and Reagent Preparation
17.5.7 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.5.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.5.9 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 Modifictions to method 905:
- 18.1.1 Fuming nitric, instead of concentrated nitric acid, is used to remove interferences.
- 18.1.2 Strontium is precipitated as a carbonate salt rather than as a sulfate salt.
- 18.1.3 The iron hydroxide scavange step is omitted; interferences are minimized through the various precipitation steps in our procedure.

18.2 Modifications to method Sr-03-RC:
- 18.2.1 The iron hydroxide scavange step is omitted.
- 18.2.2 Strontium carrier, not Strontium-85 tracer, to determine yield.
- 18.2.3 The iron hydroxide scavange step is omitted; interferences are minimized through the various precipitation steps in our procedure.
- 18.2.4 In place of the filtration steps, the samples are centrifuged.

19.0 CHANGES TO PREVIOUS REVISION

19.1 No Changes, Annual Review

19.2 Rev. 10,
- 19.2.1 Added Sr-02 and Stronium-90 to sections 1.0 and 17.0.

19.3 Rev. 11:
- 19.3.1 Added reference to section 18.2 regarding samples being centrifuged in place of filtered.
- 19.3.2 Updated section 6.0 by adding new equipment.
- 19.3.3 Removed strontium-89, NIST traceble from section 7.0.
- 19.3.4 Updated information regarding pH checking of samples acidified and not acidified by sample control in section 8.3.
- 19.3.5 Added instruction for leached analyses in section 9.2 and 9.3.
- 19.3.6 Updated procedure for high and low density samples in section 11.1.
- 19.3.7 Added calculation for determining the density of aqueous samples in section 12.0.

19.4 Rev. 12:
- 19.4.1 Grammatical corrections throughout
- 19.4.2 Removed 180 day holding time requirement from section 8
- 19.4.3 Updated section 15

19.5 Rev. 13:
- 19.5.1 Prcedure updated in section 11.0
- 19.5.2 Grammatical errors fixed through out SOP

Title: ISOTOPIC URANIUM BY EICHROM® UTEVA RESIN FOR VARIOUS MATRICES

Approvals (Signature/Date):

Sarah C. Bernsen 4.17.14 Sarah Bernsen Date Radiochemistry Prep Supervisor	Michael Ridenhower 4/17/14 Michael Ridenhower Date Health & Safety Manager / Coordinator
4-16-14 Tony L. Byrd Date Quality Assurance Specialist	Elaine Wild 4/16/14 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0238 Rev. 14

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1.0 SCOPE AND APPLICATION

1.1 This SOP provides a separation method for the measurement of uranium in water, soil, sludge and filter samples.

1.2 This method is based on Eichrom Technologies, Inc. Analytical Procedures "ACS07 Uranium in Soil (2 g sample)", and "ACW01 Uranium and Thorium in Water".

1.3 This procedure is applicable to water, soil, filter, biota, and oil.
1.3.1 Soil, filter, biota and oils are pre-prepared in accordance with SOP, ST-RC-0004.
1.3.2 Water preparation is contained within this SOP.

1.4 The requested limits, minimum detection amounts and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP describes the method for separation of Uranium using Eichrom resin prior to measurement by alpha spectrometry. A calcium phosphate precipitation technique is used to concentrate and remove actinides from water samples. Soils, Sludge and Filters are prepared for analysis using ST-RC-0004. Tracers are used to monitor chemical recoveries and yield-correct analyte results.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualififers.

4.0 INTERFERENCES

4.1 Very high levels of phosphate in a water sample may cause an interference. This is indicated by a large quantity of precipitation formed. Reducing the amount of phosphate added to coprecipitate the actinides may be necessary in these cases.

4.2 Polonium-210 decays in the uranium-232 region, and can cause high tracer recoveries if not removed from the cartridge/column, using a 25 mL 3 M HNO_3 wash after the load solution has run through the cartridge/column.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH_3)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Aluminum nitrate	Oxidizer	2 mg/m^3 (TWA) Soluble salts as Al	Inhalation symptoms may include coughing, shortness of breath. Symptoms of skin contact include redness, itching, and pain. Eye contact causes irritation, redness, and pain.
Calcium nitrate	Oxidizer	None established	Inhalation symptoms include coughing and shortness of breath. Skin contact symptoms include redness, itching, and pain. Eye contact causes irritation, redness and pain.
Hydrochloric Acid	Poison Corrosive	5 ppm (Ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
Oxalic Acid	Corrosive	1mg/m^3 (TWA) 2mg/m^3 (STEL)	Inhalation symptoms include severe irritation and burns of nose, throat, and respiratory tract. Ingestion symptoms include burns, nausea, severe gastroenteritis and vomiting. Skin contact causes severe irritation and burns. Oxalic Acid is an eye irritant.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Beakers, 150-2000 mL

6.2 Analytical balance - 0.0001 g sensitivity

6.3 Centrifuge

6.4 Centrifuge tubes, poly, 50 mL with cap

6.5 Pipets, glass or plastic, disposable

6.6 Pipetter, mechanical

6.7 Fume hood

6.8 Hotplate with stirrer

6.9 Vortex mixer

6.10 pH strips, narrow range

6.11 Vacuum Box, Eichrom part number AC-24-BOX, or equivalent

6.12 Syringe filter, 25 mm acrodisc, 0.45 or 0.70 µm

6.13 Cartridge reservoirs/syringe/funnel-20 mL B-D Luer Lok syringe Part Number 301625 (Fisher part number 14-823-2B), or equivalent.

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 DI Water, obtained from the Milli-Q unit.

7.3 Ammonium hydrogen phosphate (3.2 N):

7.3.1 Dissolve 104 g of $(NH_4)_2HPO_4$ in 200 mL of water, heating gently to dissolve. Dilute to 250 mL with water and mix well.

7.4 Ammonium hydroxide (NH_4OH), reagent.

7.5 Bromoresol Purple indicator:
7.5.1 Disso1ve 0.20 g of bromocresol purple (520.24 F.W.) in 250 mL of water, add one mL of concentrated ammonium hydroxide.

7.6 Calcium nitrate (1.25 M):
7.6.1 Dissolve 51 g of $Ca(NO_3)_2$ in 100 mL of water and dilute to 250 mL with water.

7.7 Hydrochloric acid (12 M) - concentrated HCl (sp gr 1.19)
7.7.1 Hydrochloric acid (9 M) - Add 1500 mL of concentrated HCl (sp gr 1.19) to 200 mL of water and dilute to 2 L with DI water.
7.7.2 Hydrochloric acid (1.0 M) - Add 167 mL of concentrated HCl (sp gr 1.19) to 200 mL of water and dilute to 2 L with DI water.

7.8 Hydrochloric acid (5 M) - 0.05 M oxalic acid solution:
7.8.1 Dissolve 12.6 g of oxalic acid dihydrate in approximately 800 mL water. Add 834 mL of concentrated hydrochloric acid. Dilute to 2 L, add a stir bar and place on stir plate until oxilate is completely dissolved.

7.9 Nitric acid (16 M) - concentrated HNO_3 (sp gr 1.42)

7.9.1 Nitric acid solution (3.0 M) - Add 382 mL of concentrated HNO_3 (sp gr 1.42) to 1500 mL of water and dilute to 2L with DI water.

7.10 Load solution [nitric acid (3 M) - aluminum nitrate (1 M)}:

7.10.1 Weigh 1500 g $Al(NO_3)_3 \bullet 9H_2O$ in a 4-L beaker. Add 800 mL of water (first) and 764 mL of concentrated nitric acid. Dilute to 4 L with water. Add stir bar, cover with watch glass and place on stir plate until aluminum nitrate is dissolved.

7.11 Uranium-232 standard, 10-20 dpm/mL.

7.12 Natural Uranium standard, approximately 10-20 dpm/mL.

7.13 UTEVA Resin - pre-packed, 100-150 mm resin or small particle size (50-100 mm) in appropriate plastic cartridge.

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

8.3.1 The pH of aqueous samples are checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis.

8.3.2 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior to analysis.

8.4 Solid sample requirements are found in SOP ST-RC-0004.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual “sample batch” does

not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water acidified with Nitric acid.

9.2.4 For non-aqueous method blanks, see soil preparation SOP, ST-RC-0004 for details.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water acidifed with Nitric acid and fortified with UNAT.

9.3.4 For soils, See soil preparation SOP, ST-RC-0004 for details.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate (SD)**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and pipette calibrations must be checked daily when used. Refer to SOP ST-QA-0005.

11.0 PROCEDURE

11.1 **For NON-AQUEOUS matrices (soil, oil, biota, etc) see SOP: ST-RC-0004 for initial sample preparation and proceed to section 11.5 of this SOP.**

11.2 Water Sample Preparation:

11.2.1 Aliquot 500 to 1000 mL of the sample (or enough to meet required detection limit) into an appropriate size beaker.

11.2.1.1 If a sample is known or supspected (based upon client knowledge, project scope or site history) to have a high density (>1.2 g/mL, e.g. a brine or waste) or low density (<0.98 g/mL, e.g. mixed solvent) the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).

11.2.2 Prepare method blank and LCS using 500 mL or 1000 mL DI water (match to volume of associated samples).

11.2.2.1 Acidify with nitric acid to a pH < 2.

11.2.2.2 Typically 500 mL for short count and 1000 mL for long count

11.2.3 Add appropriate tracers or standards. Generally 10 – 20 dpm of the Uranium tracer is added

11.2.3.1 Spike LCS and MS (if applicable) with isotopes of interest.

11.3 Evaporation (Alternative option to Calcium Phosphate precipitation):

11.3.1 This option may be used when large sample volumes are needed to achieve low level reporting limits.

11.3.1.1 Consult Manager/Supervisor to determine when this option should be used.

11.3.2 Evaporate sample on a hot plate to less than 50 mL and transfer to a 100 mL beaker.

11.3.2.1 Note: For some water samples, calcium sulfate formation may occur during evaporation. If this occurs, use the calcium phosphate precipitation option in step 11.4

11.3.3 Gently evaporate the sample to dryness and redissolve in approximately 5 mL of concentrated HNO_3 (sp gr 1.42). Repeat step two more times, evaporate to dryness and proceed to step 11.5.

11.4 Calcium phosphate precipitation:

11.4.1 Add 0.5 mL of 1.25M $Ca(NO_3)_2$ to each beaker.

11.4.2 Add 0.200 mL of 3.2 M $(NH_4)_2HPO_4$ solution to each beaker

11.4.3 Add 3-5 drops Bromocresol Purple indicator to each beaker.

11.4.4 Place each beaker on a hotplate.

11.4.5 Allow the samples to heat to near boiling approximately 30 minutes.

11.4.6 Once the samples reach near boiling turn the heat down to medium.

11.4.7 While stirring, add enough concentrated NH_4OH with a squirt bottle to reach the bromocresol purple indicator end point and form $Ca_3(PO_4)_2$ precipitate.

11.4.8 Allow the sample to heat uncovered for another 20-30 minutes.

11.4.9 Remove from the hot plate, allow sample to cool and precipitate to settle.

11.4.10 Decant.

11.4.11 Transfer the precipitate to a centrifuge tube using DI water and centrifuge the precipitate for approximately 5 minutes at 2000 rpm.

11.4.12 Decant supernatant and discard to waste.

11.4.13 Proceed to section 11.5.

11.5 Separation of U from other actinides using UTEVA resin

11.5.1 Dissolve calcium phosphate precipitate, soil dissolution residue or evaporated water sample with 15 mL of Load solution. Heat as necessary.

Note: Additional 5 mL load solution aliqouts may be necessary to dissolve the sample residue. Do not use more than 30 mL of load solution.

11.5.2 If particulates are observed, or the solution is cloudy, centrifuge the solution on high for 5 minutes. The precipitate is discarded.

11.5.3 The use of filtration is also permitted, e.g. syringe filter if the solution is still cloudy.

11.5.4 For each sample dissolved in load solution, place a UTEVA cartridge into the vacuum box. Place an empty centrifuge tube into the holder inside the vacuum box to catch the solution. Attach a plastic syringe funnel to the top of the cartridge.

11.5.5 Just prior to loading the sample, add 5 mL of 3 M HNO_3 into each funnel. Turn on the vacuum pump and attach it to the vacuum box. Allow the solution to be pulled through the column. This will condition the resin. The flow rate should be approximately 3 mL per minute.
Approximately 20 drops equals 1 mL. Use the valve to adjust the flow for each individual sample. Adjust the flow for each solution added.

11.5.6 Transfer each sample solution into the appropriate UTEVA resin cartridge funnel. The flow rate should be approximately 1 mL per minute.

11.5.7 Rinse the cartridge with 25 mL of 3M HNO_3. The flow rate should be approximately 3 mL per minute.

11.5.7.1 **NOTE: This will remove polonium-210 which is a potential interference at the U-232 energy.**

11.5.8 Add 5 mL of 9 M HCl into each cartridge/column and allow to pass through. The flow rate should be approximately 1 mL per minute.

11.5.8.1 **NOTE: This rinse converts the resin to the chloride form. Some Np may be removed here**.

11.5.9 Add 20 mL of 5 M HCl-0.05 M oxalic acid solution into each cartridge/column and allow it to pass through. The flow rate should be approximately 1 mL per minute.

11.5.9.1 **NOTE: This rinse removes neptunium and thorium from the cartridge/column. The 9 M HCl and 5 M HCl-0.05 N oxalic acid rinses, also remove any residual ferrous ions.**

11.5.10 Discard the waste in the collection centrifuge tube. Place a labled 50-mL centrifuge tube under each cartridge.

11.5.11 Add 15 mL of 1.0 M HCl into each cartridge to strip the uranium. The flow rate should be approximately 1 mL per minute. Collect eluant.

11.5.12 To co-precipitate the uranium, proceed to ST-RC-0100.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Density: sample weight divided by the volume of said sample

12.1.1 $d = m/v$

d = density
m = mass
v = volume

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis QAM. Specific analysis calculations are given in the applicable analytical SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the associated analytical SOP

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Eichrom Technologies, Inc. Analytical Procedures “ACS07 Uranium in Soil (2 Gram Sample)”, March 2002.

17.2 Eichrom Technologies, Inc. Analytical Procedures “ACW01 Uranium and Thorium in Water”, April 2001.

17.3 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

17.4 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.6 Associated SOPs (current revisions)

- 17.6.1 ST-PM-0002 Sample Receipt and Chain of Custody
- 17.6.2 ST-QA-0002, Standards and Reagent Preparation
- 17.6.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
- 17.6.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
- 17.6.5 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
- 17.6.6 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
- 17.6.7 ST-RC-0100, Actinide Coprecipitation
- 17.6.8 ST-RC-5006, Decontamination of Laboratory Labware, Labware and Equipment
- 17.6.9 ST-RD-0210, Daily Operations of an Alpha Spectroscopy System (using AlphaVision Software)

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD.

18.1 Bromocresol purple indicator is used throughout, where as the Eichrom method switches from bromocresol purple to phenolphthalien indicator in mid-process.

19.0 CHANGES FROM PREVIOUS REVISON

19.1 Updated standards and reagents in section 7.0

19.2 Rev. 12:

- 19.2.1 Updeated section 11.4 changed the amount of time a sample is spun in a centrifuge from ten minutes to five
- 19.2.2 Added “Heat as necessary” step to section 11.5.1.

19.3 Revision 13:

- 19.3.1 Updated section 7.0 regarding preparations for a hydrochloric acid standard and reagent mix.
- 19.3.2 Added instructions for checking samples pH in section 8.3.
- 19.3.3 Updated section 11.0 regarding water sample preparation and calcium phosphate precipitation.

19.4 Revision 14:

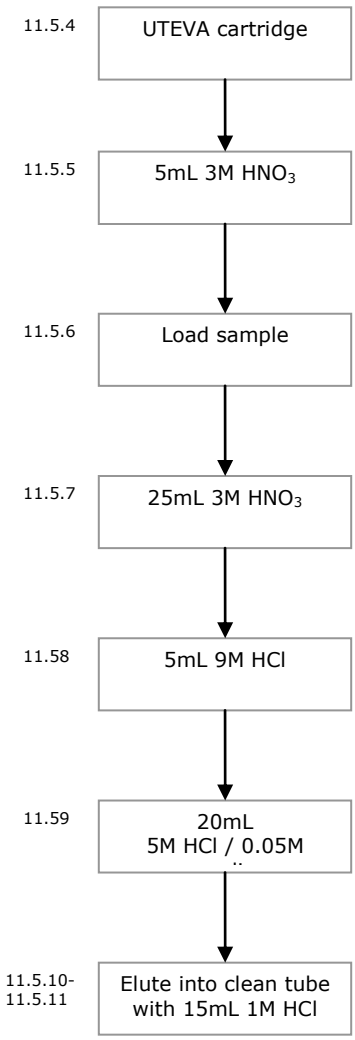
- 19.4.1 Removed QuantIMS and replaced with LIMS (Laboratory Information Management System) throughout SOP.
- 19.4.2 Updated water sample prep throughout section 11.0.
- 19.4.3 Adding density calculation to section 12.0.
- 19.4.4 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.

19.5 Revision 15:

19.5.1 Updated section 7.14 deleted TRM reference material
19.5.2 Updated section 9.3, deleted solid reference material used for soil LCS
19.5.3 Updated section 11.4 deleted stir samples
19.5.4 Updated grammatical errors through out SOP

Uranium only via UTEVA

*All rinses should flow at 1mL/minute (only 3M HNO3 may be done at 3 mL/minute)



Title: GAMMAVISION® ANALYSIS

Approvals (Signature/Date):

Chris Hough — 4/17/14 Chris Hough — Date Radiochemistry Manager	Michael Ridenhower — 4-18-14 Michael Ridenhower — Date Health & Safety Manager / Coordinator
4-15-14 Tony L. Byrd — Date Quality Assurance Specialist	Elaine Wild — 4/15/14 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-RD-0102 Rev. 11

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1.0 SCOPE AND APPLICATION

1.1 This procedure applies to all germanium detectors and the computer assisted germanium spectroscopy analysis system.

1.2 Due to the nature of gamma spectroscopy, once the system is calibrated to a particular geometry a similar matrix may be run as long as it is prepared to match a calibrated geometry.

1.3 This SOP is based on EPA Method 901.1 and DOE EML HASL 300 Method GA-01-R.

1.4 The requested limits (**RL**), minimum detectable amount (**MDA**) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This procedure provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the germanium spectroscopy system.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Germanium spectrometry has potential interference. Interferences are usually in the form of radionuclides with unresolved photon emissions. These interferences are limited by the careful design/construction of the gamma spectral identification and interference libraries.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6.0 EQUIPMENT AND SUPPLIES

6.1 Germanium spectroscopy system utilizing a computer based data acquisition system (GammaVision®-32).

6.2 GammaVision®-32 (know as GammaVision) is a comprehensive, all-in-one package, for the analysis of gamma-ray spectra acquired with HPGe detectors.

6.3 Global Value software is an optimization tool for automation, custom reporting, quality assurance and data management (GammaVision productivity add-on software).

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 Commercially prepared mixed gamma standards in reproducible geometries, with all appropriate NIST Source Certificate information.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Samples may be collected in glass or plastic containers.

8.2 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a <u>method blank</u>, a <u>Laboratory Control Sample</u> , and <u>Sample Duplicate</u>.

9.3 **Method Blank (MB)**

9.3.1 A method blank must be counted with every sample batch.

9.3.1.1 For soils, a method blank is sodium sulfate filled in the specified geometry.

9.3.1.2 For waters, a method blank is DI water filled in the specified geometry.

9.3.1.3 For filters, a method blank is a blank petri dish.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 An LCS must be counted with every sample batch.

9.4.1.1 For water, a purchased mixed nuclide source in the specified geometry.

9.4.1.2 For soil, a purchased mixed nuclide source in the specified geometry.

9.4.1.3 For filters, a purchased mixed nuclide source in a petri dish.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is a recounted field sample to demonstrate instrument precision, since there is no sample preparation (required to count on a different detector than the sample).

9.5.1.1 If requested, the laboratory may perform a Sample Duplicate which is an additional aliquot of a field sample.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 There are two types of Calibrations performed for Gamma: Energy and Efficiency

10.1.1 Energy Calibrations

10.1.1.1 Frequency: the energy calibration is performed once per detector. The source is not geometry specific.

10.1.1.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include significant changes in instrument operating parameters, and major instrument maintenance (e.g. replacing the detector)

10.1.1.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.1.1.4 Range: the energy range, is 46.54 to 1836.1 keV for air filter and solid.

10.1.1.5 Criteria:

10.1.1.5.1 The curve should have, at minimum, eight calibration points used to determine the energy relationship of the calibration.

10.1.1.5.2 The energy difference (delta Δ) should be within 0.05% for all calibration points or within 0.2 keV for the calibration points.

10.1.1.5.3 The FWHM must be less than 3.0 keV at 1332 keV.

10.1.1.5.4 FWHM difference (delta Δ) should be within 8% for all calibration points.

10.1.2 Efficiency Calibrations

10.1.2.1 Frequency: the efficiency calibration is performed per geometry.

10.1.2.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include significant changes in instrument operating parameters, and major instrument maintenance (e.g. replacing the detector)

10.1.2.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.1.2.4 Range: the energy range of the calibration is dependent on the matrix that is calibrated. i.e. 46.54 to 1836.1 keV for air filter and solid, 59.5 keV to 1836.1 keV for water.

10.1.2.5 Criteria:

10.1.2.5.1 The curve should have at least eight points to determine the efficiency

10.1.2.5.2 The calibration source must have radionuclides that “bracket” the intended range of calibration

10.1.2.5.3 A minimum of 10,000 counts will be accumulated for each data point

10.1.2.5.4 The efficiency difference (delta Δ) should be within 8% of the true value for each point

10.2 Initial Calibration Verification (ICV) [Frequency: Once]

10.2.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.2.1.1 The ICV check does not include short half-life nuclides which may exist in the purchased standard. At a minimum, the ICV will always contain Am-241 (low), Cs-137 (medium) and Co-60 (high).

10.2.2 An ICV must be performed with every initial calibration.

10.2.3 The ICV percent recovery must be within ± 10% of the true value for each nuclide.

10.2.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a

different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.3 Annual Calibration Verification (ACV) [Frequency: Annually] not geometry specific

10.3.1 An annual calibration verification check will be performed on each detector.

10.3.2 Two verification standards (second source independent from the initial calibration source) will be used for the verification checks.

10.3.2.1 One from a water source that surrounds the detector

10.3.2.2 One from a solid source that rests on top of the detector

10.3.3 The checks will include isotopes from the low (Am-241), medium (Cs-137) and high (Co-60) energy range.

10.3.4 The verification can be accomplished by using LCS samples counted on each detector.

10.3.5 The ACV percent recovery must be within ± 10% of the true value for each nuclide.

10.3.6 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ACV, or analysis of a different ACV). Any decision to proceed with analysis of samples when the ACV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.4 Daily Checks

10.4.1 The detector **background** shall be checked each day that the germanium spectroscopy system is used. Limits are set at 2 sigma and 3 sigma.

10.4.1.1 <u>Bkgd Countrate (background count rate for entire spectrum)</u>

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.4.2 The instrument **Channel**, **Energy**, **FWHM** (resolution) and **Activity Difference** (efficiency) for a detector shall be checked each day the germanium spectroscopy system is used (using a check source that is non-geometry specific).

10.4.2.1 **Channel** – (low and high energy) is monitored for channel alignment. Limits are set around the target Channel

10.4.2.1.1 QA-60 <u>Low Energy</u>

Tolerance (warning)	=	± 1 channel
Control (out)	=	± 2 channels

10.4.2.1.2 QA-1332 <u>High Energy</u>

Tolerance (warning)	=	± 2 channels
Control (out)	=	± 3 channels

10.4.2.2 **Energy** – (low and high energy) is monitored for energy alignment. Limits are set around a target energy

10.4.2.2.1 QA-60 <u>Low Energy</u>

Tolerance (warning)	=	± 0.25 keV
Control (out)	=	± 0.50 keV

10.4.2.2.2 QA-1332 <u>High Energy</u>

Tolerance (warning)	=	± 0.5 keV
Control (out)	=	± 0.75 keV

10.4.2.3 Full-Width at the Half Maximum (**FWHM**) - (low, mid, and high energy) is monitored for peak shape There are no limits compared to a target FWHM. There are no lower limits (–) set for FWHM.

10.4.2.3.1 QA-60 <u>Low Energy</u>

Tolerance (warning)	=	+ 1.1
Control (out)	=	+ 1.2

10.4.2.3.2 QA-662 <u>Mid Energy</u>

Tolerance (warning)	=	+ 1.7
Control (out)	=	+ 1.8

10.4.2.3.3 QA-1332 <u>High Energy</u>

Tolerance (warning)	=	+ 2.2
Control (out)	=	+ 2.3

10.4.2.4 **Activity Difference** (low, mid, and high energy) – is monitored to check the percent difference between the source activity and the reported activity. Limits are set around the target activity.

10.4.2.4.1 QA-60/662/1332 <u>Low/Mid/High Energy</u>

Tolerance (warning)	=	± 4
Control (out)	=	± 5

10.4.3 If the daily check is outside of the control limits, it may be recounted or tagged out with red tag (or with an NCM). The Daily QC check may only be recounted once without corrective action.

10.4.3.1 If the out of control parameter is found acceptable for the rerun, the instrument can be used for the analysis of samples. ***Note***: *No corrective action is necessary for this situation since the uncertainty can be attributed to the stochastic uncertainty of decay process (statistics), uncertainty of the sources, or a known uncorrected trend.*

10.4.3.2 If the instrument fails to meet the acceptance criteria for the rerun, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out". (See ST-QA-0036 for NCM details regarding tagging out of service).

10.4.3.3 If the QC check fails the following day for the same detector for the same specific parameter as the day before, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out" until the detector can be evaluated and/or maintenance can be performed.

10.4.3.4 The analyst may want to:

10.4.3.4.1 Check the expiration date of the radioactive standard to confirm the material is current, for the isotopes being utilized.

10.4.3.4.2 Check source positioning and all instrument settings.

10.4.3.4.3 Check all cables for any apparent damage and confirm that all cables are routed to proper connectors and are in good working order.

10.4.3.4.4 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Corrective actions must be noted in the instrument maintenance log.

10.4.3.4.5 If a parameter has two successive values in the warning limits, the system will be examined for a trend and noted in the maintenance log. Decisions will be based upon the Data Quality Objectives (DQO) and the degree of the bias in relation to the parameter.

10.5 Background

10.5.1 Background subtraction spectrum shall be established for the germanium spectroscopy systems **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements.

10.5.1.1 Background count time is 12 hours.

10.5.1.1.1 If a client project requires a longer count time, then the background must be performed at the longer time before initiating analysis.

10.5.1.1.2 After review of the monthly background, the analyst will mark each detector complete on the “Monthly Background Complete” sheet located on each gamma cave (see attachment 2)

10.5.1.2 Monthly Background limits are set at 2 sigma and 3 sigma.

10.5.1.2.1 <u>Bkgd Countrate (background count rate for entire spectrum)</u>

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.6 Calibration Software Handling

10.6.1 Gamma Detector System Energy and Shape Calibration

10.6.1.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:

10.6.1.1.1 X = Detector Number
10.6.1.1.2 Analysis method
10.6.1.1.3 Select library
10.6.1.1.4 Enter correct sample data.
10.6.1.1.5 Enter correct conversion time.

10.6.1.2 Close all detectors windows in the current instance of gamma vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.1.3 Select the menu "Analyze\Setting\Sample type…."

10.6.1.4 Select the browse button next to the "File" field and open the file. Click the "OK" button of the window to close it.

10.6.1.5 Recall the application Calibration File from the menu "Calibration \Recall Calibration…"

10.6.1.6 Select the menu "Calibrate\Calibration wizard…"

10.6.1.7 Select the option to create new energy calibrations. Select the next button.

10.6.1.8 On the energy calibration wizard page, select the file "DET_EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the next button.

10.6.1.9 Select the next button to perform the energy, FWHM.

10.6.1.10 Select the edit energy button to review the energy.

10.6.1.10.1 Close the energy calibration sidebar window.

10.6.1.11 Select the save calibration button and save the calibration to "Cal\Energy\X_Energy.clb" where X is the detector.

10.6.1.12 Enter the calibration description in the format "X_ENERGY_GEOMETRY" where X is the detector number and Geometry is an appropriate geometry description when prompted. Select the Finish button to close the calibration wizard.

10.6.1.13 Print the calibration report from the menu "Calibrate \print calibration.

10.6.2 Gamma Detector System Efficiency Calibration

10.6.2.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:

10.6.2.1.1 X = Detector Number
10.6.2.1.2 Analysis method
10.6.2.1.3 Select library
10.6.2.1.4 Enter correct sample data.
10.6.2.1.5 Enter correct conversion time.

10.6.2.2 Close all detector windows in the current instance of Gamma Vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.2.3 Select the menu "Analyze\Setting\Sample Type"

10.6.2.4 Select the browse button next to the "File", field and open the file. Click the "OK" button at the bottom of the window to close it.

10.6.2.5 Recall the applicable calibration file from the menu "Calibration\Recall Calibration" (if the geometry file currently exists)

10.6.2.6 Select the menu "Calibrate\Calibration Wizard"

10.6.2.7 Select the option to create new energy and efficiency calibration. Select next button.

10.6.2.8 On the Energy Calibration Wizard page select the file "EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the Next button.

10.6.2.9 On the Efficiency Calibration Wizard page, select library file, "DET_EfficiencyCalibration.Lib" for mixed gamma sources.

10.6.2.10 On the Efficiency Calibration Wizard page, select the appropriate Certification file from the directory.

10.6.2.11 Select the next button to perform the energy FWHM and efficiency calibration.

10.6.2.12 Select the Edit Energy button to review the energy and FWHM Calibration.

10.6.2.12.1 Close the Efficiency Calibration side window.

10.6.2.13 Select the save calibration button and save the calibration to Cal\X_Geometry.clb" where X is the detector and geometry is an appropriate geometry name.

10.6.2.14 Enter the calibration description in the format "x_Geometry_Source number_date counted" where X is the detector number and geometry is an appropriate geometry description when prompted. Select the finish button to close the calibration wizard.

10.6.2.15 Print calibration report from the menu "Calibrate\Print Calibration"

10.6.2.16 Select "Analyze", select "Entire spectrum in memory" and file print.

10.6.2.17 Close the spectrum Buffer window and save the spectrum when prompted.

10.6.3 Detector Long Background Counting

10.6.3.1 Remove any samples from the detector, clean the detector, close the shield lid and start acquisition.

10.6.3.2 Select detector 1 in Global Value Quick Start

10.6.3.3 Select Monthly Background PBC under Automation Groups

10.6.3.4 Select Background PBC Long Count under Automation Jobs.

10.6.3.5 Login using name and password.

10.6.3.6 Select "OK", ensure detector cave is empty.

10.6.3.7 Repeat for each detector which background you would like to start.

10.6.3.8 After the background is complete it will save as a PBC file.

11.0 PROCEDURE

11.1 Calibration Quality Control (**Daily Check**)

11.1.1 Place the calibration quality control sample on the detector, and start acquisition.

11.1.2 Select detector from Global Value Quick Start.

11.1.3 Select Quality Control under Automation Groups.

11.1.4 Select Daily Quality Control Check under Automation Jobs.

11.1.5 Login with user name and password.

11.1.6 Select "OK", ensure source is on detector.

11.1.7 Repeat for each detector.

11.1.8 Record in the instrument run log.

11.2 Background Quality Control (**Daily Background**)

11.2.1 Remove any samples from the detector, and start acquisition.

11.2.2 Select detector global value quick start.

11.2.3 Select quality control under automation groups.

11.2.4 Select daily background check under automation jobs.

11.2.5 Login with username and password.

11.2.6 Select "OK", ensure detector cave is empty.

11.2.7 Repeat for each detector.

11.2.8 Record in the instrument run log.

11.3 Sample Counting

11.3.1 Place the sample on the detector.
11.3.2 Select detector from GlobalValue quick start.
11.3.3 Select analyze samples under automation groups.
11.3.4 Select count sample under automation jobs.
11.3.5 Login with username and password.
11.3.6 Scan sample description from barcode report.
11.3.7 Select analysis method, sample type, geometry, library, correct date, count time, continue.
11.3.8 Select "OK", ensure sample is on detector.
11.3.9 Record in the instrument run log.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 All calculations are performed in GammaVision-32 software; conversions are performed in RadCapture. Calculations are found in ST-QAM.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (**MB**)

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the requested limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 <u>Method Blank Contamination</u> – The blank may be re-counted once to confirm the activity (in the same detector). If the re-counted MB activity exceeds the MDA and/or the requested limit, samples with less than 10 times the activity found in the blank are recounted. An NCM is written to document the excursion. Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in LIMS.

13.3 Laboratory Control Sample (**LCS**)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 <u>LCS Spike Recovery excursion (high)</u> – The LCS may be re-counted once to confirm the result. If the re-counted LCS exceeds the control limit, samples that are non-detect may be reported with an NCM.

13.3.2.2 <u>LCS Spike Recovery excursion (low)</u> – The LCS may be re-counted once to confirm the result. <u>If the low recovery is confirmed, the batch is recounted</u>.

13.3.2.3 RPD/RER Duplicate excursion – The LCS is recounted if both RPD and RER exceed criteria.

13.4 Duplicate

13.4.1 Acceptance Criteria:

13.4.1.1 All control analytes must be within the specified control limits for precision (RPD), max. 40% RPD, RER < 1.

13.4.2 Corrective Action for Duplicate not meeting acceptance criteria:

13.4.2.1 RPD/RER Duplicate excursion – The sample is recounted if both RPD and RER exceed criteria.

13.5 Insufficient Sample

13.5.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. The insufficient sample description is included in the LIMS NCM within the type defining the excursion.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration of capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for “Waste Management and Pollution Prevention.”

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 28th Edition, method GA-01-R, Gamma Radioassay
17.2 EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method 901.1
17.3 American National Standards Institute (ANSI) Accredited Standards Committee on Radiation Instrumentation, N42; ANSI N42.14-1999, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides
17.4 Ortec MCB Connections-32, Hardware Property Dialogs Manual, current version
17.5 MAESTRO–32, MCA Emulator, current version
17.6 GammaVision–32, Gamma-Ray Spectrum Analysis and MCA Emulator, current version
17.7 Master library Source: Gerhard Erdtmann, Werner Soyka
17.8 TestAmerica Quality Assurance Manual (ST-QAM), current revision
17.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
17.10 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
17.11 Associated SOPs, Current Revision:
- 17.11.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
- 17.11.2 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
- 17.11.3 ST-RC-0025, Preparation of Samples for Gamma Spectroscopy
- 17.11.4 ST-QA-0002, Standards and Reagent Preparation
- 17.11.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.11.6 ST-QA-0036, Non-Conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Annual Review, No Changes.
19.2 Revision 8:
- 19.2.1 Increased background count times from 12 to 36 hours in section 10.3.1.1.
- 19.2.2 Updated the procedure for detector long background counting in section 10.5 to reflect new software.
- 19.2.3 Updated daily calibration checks, daily background and sample counting procedures in section 11.0 to reflect new software.

19.3 Revision 9:
- 19.3.1 Replaced quartz sand with sodium sulfate to be used for soil method blanks in section 9.2.
- 19.3.2 Updated section 10.4 regarding instrument daily checks.
- 19.3.3 Updated data assessment and acceptance criteria in section 13.0
- 19.3.4 Updated section 9.0 regarding batch, method blank and laboratory control samples.
- 19.3.5 Updated the calibation points for an internal calibation in section 10.1.
- 19.3.6 Updated the percent recovery regarding the ICV in section 10.2.
- 19.3.7 Updated software storage file name throughout section 10.5.

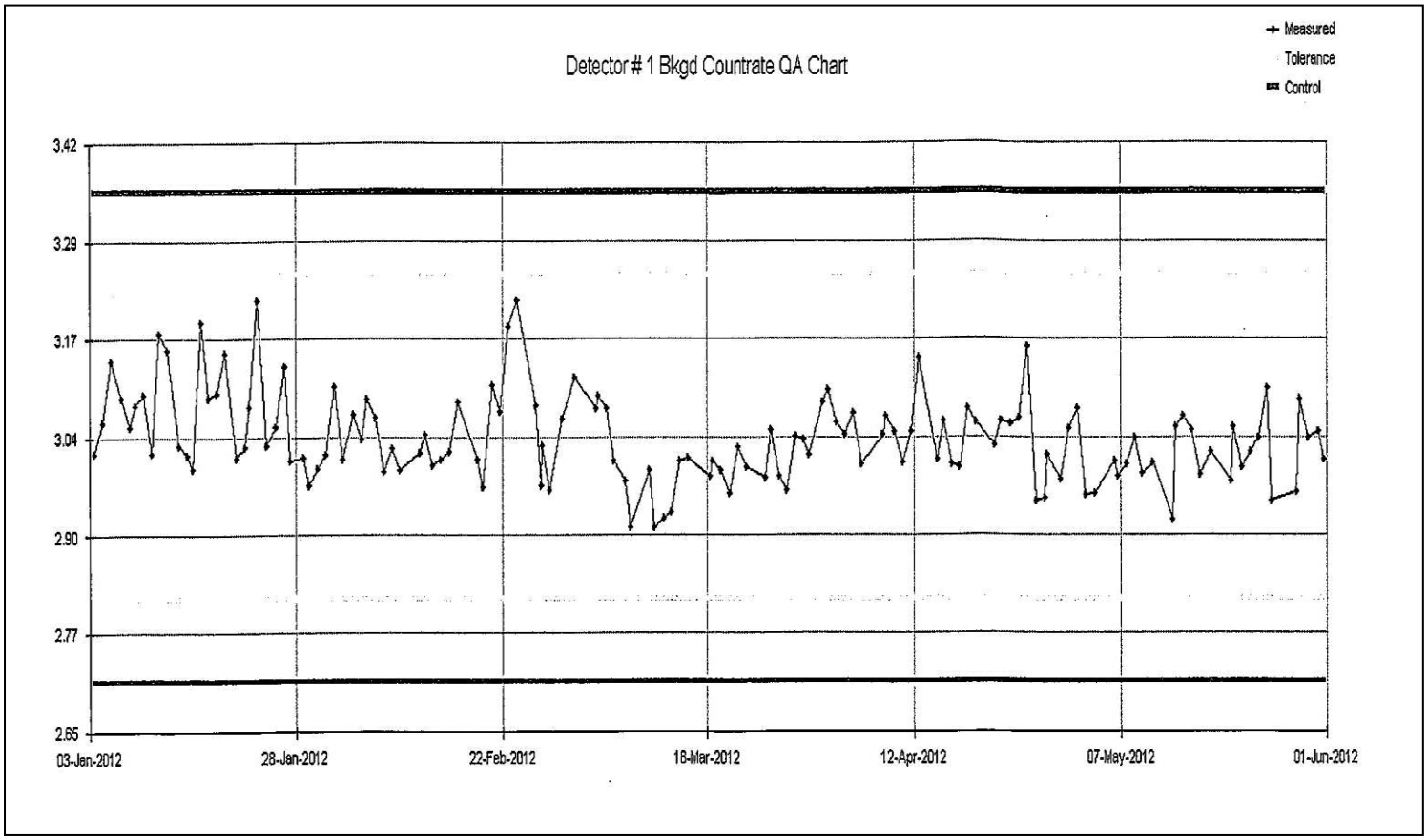
19.4 Revision 10:
- 19.4.1 Updated references to QuantIMS through out
- 19.4.2 Update §10.1
- 19.4.3 Added §10.3 Annual Calibration Verification
- 19.4.4 Updated §10.4: 36 hour background changed to 12 hour and requirement to complete Attachment 2

19.4.5 Added Attachment 2, "Monthly Background Complete" example
19.4.6 Updated §13 references to Clouseau changed to LIMS
19.4.7 Added §17 reference to ANSI 42.14-1999

19.5 Revision 11:
19.5.1 Updated §1.4 with corrected termonolgy
19.5.2 Updated §6.0 software details
19.5.3 Additon/Update §10.0 major change in calibration
19.5.4 Updated §13.0 additional corrective action steps
19.5.5 Updated §15.0 with new verbiage

19.6 Revision 12:
19.6.1 Spelling and grammar corrections made throughout SOP.
19.6.2 Sections 10.2.3 and 10.2.5 had wording changed to common text.
19.6.3 Section 10.4.3.3 was updated to add 'for the same specific parameter as the day before' and 'until the detector can be evaluated and/or maintenance can be performed.'.
19.6.4 Section 10.5.1.2.1 was added to provide limits for monthly backgrounds, which were not previously provided.
19.6.5 Section 13.4.2 had 'LCS' changed to 'duplicate' since it is the duplicate section and LCS was incorrectly referenced.

Attachment 1



Attachment 2

Example of the form, actual form in use may have slight variations.

2013 Monthly Background Complete

Jan	Reveiwed by_____ Initials & Date
Feb	Reveiwed by_____ Initials & Date
Mar	Reveiwed by_____ Initials & Date
April	Reveiwed by_____ Initials & Date
May	Reveiwed by_____ Initials & Date
June	Reveiwed by_____ Initials & Date
July	Reveiwed by_____ Initials & Date
Aug	Reveiwed by_____ Initials & Date
Sept	Reveiwed by_____ Initials & Date
Oct	Reveiwed by_____ Initials & Date
Nov	Reveiwed by_____ Initials & Date
Dec	Reveiwed by_____ Initials & Date

Title: ALPHA SPECTROSCOPY ANALYSIS [DOE HASL-300 A-01-R]

Approvals (Signature/Date):

Chris Hough 4/24/15 Date Radiochemistry Manager	Michael Ridenhower 4/24/15 Date Health & Safety Manager / Coordinator
Marti Ward 4.24.15 Date Quality Assurance Manager	Elaine Wild 4/24/15 Date Laboratory Director

This SOP was previously identified as SOP No. ST-RD-0210 Rev. 11

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1.0 SCOPE AND APPLICATION

1.1 This procedure applies to alpha spectroscopy detectors and the computer assisted alpha spectroscopy analysis systems, using AlphaVision software.

1.2 This SOP is based on DOE method A-01-R

1.3 This SOP is applicable to both liquid and solid matrices.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the alpha spectroscopy system.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for glossary of common terms and data qualifiers.

3.2 Tracer – A known amount of ^{232}U, ^{242}Pu or ^{236}Pu, ^{243}Am, ^{209}Po, ^{244}Cm or ^{229}Th (depending on analyte(s) required) added to each sample to determine chemical yield. The tracer serves as an internal standard, which is used to calculate the activity of the target isotopes.

3.3 Region of Interest (ROI) – The keV range through which the target isotope peak signal responds.

3.4 keV - (kilo electron Volt) – electron volt is a unit of energy defined as the amount of energy gained (or lost) by the charge of a single electron moved across an electric potential difference of one volt.

3.5 Tailing – Tailing is a delayed return of a peak to chromatographic baseline or continuation of response beyond its normal response window (RT window, ROI) due to high concentration of the analyte or matrix interference.

3.6 AlphaVision – The Alpha Spectrometer data collection and processing software.

4.0 INTERFERENCES

4.1 Alpha spectrometry has many potential interferences. These are usually in the form of radionuclides with unresolved alpha emissions. Poorly resolved alpha peaks are often due to high alpha activity rates or attenuation of the alpha emissions.

4.2 Isotope peak responses, when sufficiently high, may tail into other isotope ROI. Th-229 tailing into the Th-230 region of interest is a recognized example. This interference is minimized by maintaining low activities of the Th-229 tracer and monitoring of the separation of the ROI for Th-229 and Th-230. The use of manual integration may be required.

4.3 Some isotopic elements are not distinguishable and are reported as an isotopic pair, unless specifically directed by the client not to do so. These pairs may be reported separately depending

on the client's DQO and the use-ability of the data. When reported separately, the narrative must describe the technical aspects of how the isotopic pair was divided.

4.3.1 Recognized Isotopic Pairs:

4.3.1.1 Plutonium-239/240
4.3.1.2 Uranium-235/236
4.3.1.3 Uranium-233/234
4.3.1.4 Curium-245/246
4.3.1.5 Curium-247/248

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED
None.

6.0 EQUIPMENT AND SUPPLIES

6.1 Alpha spectroscopy system utilizing a computer based data acquisition system.

6.1.1 Hardware – Octete Plus and Alpha Ensemble
6.1.2 Software – AlphaVision and Radcapture

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Commercially prepared alpha standards for the isotopes Th-230, Pu-239 and Am-243.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank (MB)**

9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.3.2 A method blank must be prepared with every sample batch.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 An LCS must be prepared with every sample batch.

9.5 **Matrix Spike (MS)**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate (Dup)**

9.6.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**

9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA, see SOP ST-QA-0036 for details regarding the NCM process.

9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA, see SOP ST-QA-0036 for details regarding the NCM process.

10.0 INSTRUMENT SETUP, CALIBRATION, AND STANDARDIZATION

10.1 Initial instrument setup is performed when instrument is first installed, when a detector or chamber is changed/replaced, when a chamber is returned from the manufacturer after servicing, or other such circumstances. The following steps should be taken to ensure proper setup. Steps may be accomplished either through hardware knobs/potentiometers or through software settings (depending upon the system hardware/software). See the hardware and/or software manual to determine further detailed instructions:

10.1.1 Set the conversion gain to 1024 channels.

10.1.2 Adjust the coarse and fine gain as well as the offset to adjust the location of the three peaks of the alpha source such that the lower energy peak (Th-230 at 4688 keV) falls into channel 176, the mid-energy peak (Pu-239 at 5155 keV) falls into channel 239, and the higher energy peak (Am-241 at 5486 keV) falls into channel 283. Note that this results in 107 channels between the low energy and high energy peaks (about 7.46 keV/channel with offset

of approximately 3375 keV). Ensure each peak is within 2 channels of the desired channel before beginning energy and efficiency calibrations.

10.1.2.1 Gain adjustment:

10.1.2.1.1 Turning the knob counter-clockwise decreases the gain (decrease the value in the fine gain for software adjustment), causing the spectrum peaks to move closer to each other (smaller keV/channel slope) and toward the lower energy.

10.1.2.1.2 Turning the knob clockwise increases the gain (increase the value in the fine gain for software adjustment), causing the spectrum peaks to spread apart (larger keV/channel slope) and toward higher energy.

10.1.2.2 Offset adjustment:

10.1.2.2.1 Increasing the offset moves the peak/spectra toward the the left (lower channel number) without altering the keV/channel (slope).

10.1.2.2.2 Decreasing the offset moves the peak/spectra toward the right (higher channel number) without altering the keV/channel (slope).

10.1.3 Adjust the pulser setting such that the pulser peak is centered at about channel 222 (approximately 5 MeV).

10.2 Calibrations, see 10.6 for procedure.

10.2.1 Energy calibrations shall be performed for the alpha spectroscopy systems <u>yearly</u>, or when a calibration quality control check indicates an unacceptable change in the energy calibration parameters.

10.2.1.1 Energy Calibrations shall be performed using at least three isotopes within the energy range of 3-6 MeV.

10.2.1.2 Typical isotopes used are Th-230, Pu-239, and Am-241. The energy vs. channel slope should be less than 15 keV per channel. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Final peak energy positions of all observed isotopes shall be within ± 2.5 channels (~20 keV) of expected channel/energy (see 10.1.2). The actual energy vs. channel and the equation with the slope is not calculated. Setting the peaks to within 2.5 channels of expected will ensure calculations utilizing fixed Regions of Interest (ROI) for each isotope will provide accurate results with minimal need for manual adjustment of ROI.

10.2.1.3 Routine pulser checks and continuing calibration verifications (see below) will help control/monitor for drift.

10.2.2 Efficiency calibrations (initial calibrations or IC) shall be established for the alpha spectroscopy systems <u>yearly</u>, or when a calibration quality control check indicates an unacceptable change in the efficiency calibration parameters.

10.2.2.1 A minimum of 3000 net counts must be collected for each peak in the calibration. Calibrated efficiency should fall between 20% and 32%. Values outside this range do not constitute a failure. However, if the calibrated efficiency does fall outside this range, an evaluation of the suitability of the detector for use should be performed and documented.

10.2.3 Initial calibration verifications (ICV) shall be performed utilizing an independent second source following the initial calibration.

10.2.3.1 A minimum of 2000 net counts must be collected for each peak in the ICV. The efficiency of the ICV must fall withing 95%-105% of the initial calibration efficiency value. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 20 keV of the corresponding calibraation peak in the initial calibration (IC).

10.2.3.2 A second level review will be perfomed before detectors are placed into service and will be noted as acceptable in the electronic monthly maintenance log.

10.3 Continuing Calibration Verification (CCV)

10.3.1 A continuing calibration verification shall be performed on a monthly basis.

10.3.1.1 A minimum of 2000 net counts must be collected for each peak in the CCV. The efficiency should fall within 95%-105% of the calibrated efficiency. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 30 keV of the corresponding calibraation peak in the initial calibration (IC).

10.3.1.2 A second level review will be performed before detectors are placed into service and will be noted as accepable in the electronic monthly maintenance log.

10.4 Background subtraction spectrum (Initial Calibration Background or ICB) shall be established for the alpha spectroscopy systems monthly, or when the background quality control check indicates an unacceptable change in the daily background parameters.

10.5 Instrument Contamination Check (ICC) shall be performed weekly at minimum and after counting high activity samples (see section 11.9) . Must be a minimum four hour count duration.

10.6 Daily Checks (Pulsers)

10.6.1 Routine pulser quality control verifications are performed each day of use.

10.6.1.1 The pulser energy, peak centroid, peak resolution, peak area quality control for a detector shall be checked each day that the alpha spectroscopy system is used.

The limits for pulser centroid and pulser energy will be as below:

10.6.1.1.1 Gross counts must be within 5% of the average (20-point minimum) for each detector.

10.6.1.1.2 The peak resolution (FWHM) must fall within 10-20 keV.

10.6.1.1.3 The pulser centroid must fall within ± 5 channels of the average (20-point minimum) for each detector.

10.6.1.1.4 The pulser energy must fall within ± 40 keV of the average (20-point minimum) for each detector.

10.6.2 Routine calibration, background and pulser quality control parameters using the "Boundry" out-of-range test will be found unacceptable if the value is outside parameter tolerance.

10.6.2.1 The routine quality control check should be rerun to determine the statistical significance of the out of control parameter.

10.6.2.2 If the out of control parameter is found acceptable in the rerun, the investigation will be noted in the instrument maintenance log.

10.6.2.3 Check the integrity of the radioactive standard.

10.6.2.4 Check source positioning and all instrument settings.

10.6.2.5 Check all cables for any apparent damage and to confirm that all cables are routed to proper connectors and are in good working order.

10.6.3 If the instrument fails to meet the acceptance criteria, and the corrective actions above do not resolve the problem, the instrument must be "tagged" out of service (OOS) for the day see Attachment 1 for OOS tag example.

10.6.3.1 This is noted on the Alpa Spec Daily report by marking the report (The report will display FAIL for criteria not met). The physical detector will be marked out of service with the date and initials of the analyst performing the daily check.

10.6.3.2 If a detector fails three consecutive days for the same criteria, the detector will be taken out of service until the problem is resolved. This is done by clicking on the detector in Alphavision. Right click on the the detector, select detector properties, check the "out of service" box and fill in the description field briefly explaining the problem. Mark the detector with an OOS tag as a visual indicator of its status.

10.6.3.3 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Note any repairs in the maintenance log.

10.7 Calibration process in the Software

10.7.1 Alpha Detector System Energy and Efficiency Calibration

10.7.1.1 Print out the standards sheet for calibration
10.7.1.1.1 Location: \\slsvr01\rad\alpha\Calibration_Sources .
10.7.1.2 Load sources into the detector
10.7.1.3 In the AlphaVision software, click on the Calibration icon.
10.7.1.4 Click on the detector to be calibrated.
10.7.1.5 Select Process and then select calibration from the dropdown menu.
10.7.1.5.1 The Calibration Explorer Window will appear.
10.7.1.6 In the General Window: scan the source name;AV(detector #)-date (with year month day format YYYYMMDD).
10.7.1.7 Choose the correct source template.
10.7.1.8 Click next
10.7.1.9 In the Acquisition window, confirm count time of 140 minutes
10.7.1.10 Click next
10.7.1.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will be shelf 1) Make sure the 'Active' box is checked so the calibratoin is put in use immediately after calibration is processed.
10.7.1.12 Click next
10.7.1.13 In the Report Window, select print on completion
10.7.1.14 Click finish
10.7.1.15 When count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, Click Calibrate, click Save.
10.7.1.16 Verify the efficiency is above 20% and below 32%
10.7.1.17 Repeat for each detector
10.7.1.18 Record the calibration in the Alpha Maintenance Log.

10.7.2 ICV Procedure

10.7.2.1 Print out the standards sheet for ICV
10.7.2.1.1 Location: \\slsvr01\rad\alpha\Calibration_Sources
10.7.2.2 Place the correct source into the detector.
10.7.2.3 In The AlphaVision software, click on the Calibration Icon
10.7.2.4 Click on the detector to be calibrated
10.7.2.5 Select Process and then select calibration from the dropdown menu
10.6.2.5.2 The Calibration Explorer Window will appear.
10.6.2.6 In the General Window; scan the source name;AV(detector #)-date (with year month day format YYYYMMDD).
10.6.2.7 Chose the correct source template
10.6.2.8 Click next
10.6.2.9 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will be shelf 1)
10.6.2.10 Click next
10.6.2.11 In the Report Window, select print on completion
10.6.2.12 Click finish
10.6.2.13 When the count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, click Calibrate and click Save.
10.6.2.14 Verify the efficiency is above 20% and below 32%
10.6.2.15 Repeat for each detector
10.6.2.16 Record the ICV in the Alpha Maintenance Log.
10.6.2.17 Open the AlphaVision Access database program on computer slrad18
10.6.2.18 Select QC main from the form tab
10.6.2.19 Enter date range
10.6.2.20 Select system 1 for AlphaVision or system 2 for AlphaVision 1

10.6.2.21 Select Get Cal Data.
10.6.2.22 Exit
10.6.2.23 Select Check Ver to run the report and verify the ICV passes criteria.
10.6.2.24 For the Intitial Calibratin (IC), the detectors must have the box checked in the Cal Data window for that specific calibration and the previous year's IC must be unchecked and the 'do not use' box must be checked to ensure the correct calibration is being used.

10.7.3 CCV Procedure

10.7.3.1 Print out standards sheet for CCV on the network
10.7.3.1.1 Location: \\slsvr01\rad\alpha\Calibration_Sources
10.7.3.2 Load sources into the detectors
10.7.3.3 In the AlphaVision software, click on the Calibration Icon.
10.7.3.4 Click on the detector to be calibrated
10.7.3.5 Select process and then select calibration from the dropdown menu
10.7.3.5.1 The Calibration Explorer Window will appear.
10.7.3.6 In the General Window; scan the source name;AV(detector #)-date (with year month day format YYYYMMDD).
10.7.3.7 Select correct source template
10.7.3.8 Click Next
10.7.3.9 In the Acquisition window, confirm count time of 60 mins
10.7.3.10 Click Next
10.7.3.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used and select which shelf the source is on. (This will be shelf 1)
10.7.3.12 Click next
10.7.3.13 In the Report Window, select print on completion
10.7.3.14 Click finish
10.7.3.15 When the count is complete, the Manual Energy and Efficiency Calibration window will appear. In this window, select Calibration ROI, select Calibrate and Save.
10.7.3.16 Verify the efficiency is above 20% and below 32%.
10.7.3.17 Repeat for each detector
10.7.3.18 Record the CCV in the Alpha Maintenace Log.Efficiency must be greater than 20% and less than 32%.
10.7.3.19 Open the AlphaVision Access database program on computer slrad18.
10.7.3.20 Select QC main from the form tab.
10.7.3.21 Enter date range.
10.7.3.22 Select system 1 for AlphaVision or system 2 for AlphaVision 1.
10.7.3.23 Select Get Cal Data.
10.7.3.24 Exit.
10.7.3.25 Select Check Ver to run the report and verify the CCV passes criteria.

10.7.4 Detector Background Process (ICB) (See Section 10.4)

10.7.4.1 Select the Batch Icon
10.7.4.2 Select backgrounds from the Tool Bar
10.7.4.3 Select Process.
10.7.4.3.1 This will open the General Window in Batch Wizard
10.7.4.4 Name the background with month year format; MonthYYYY (e.g. January2015)
10.7.4.5 Select correct template (provided by analyst)
10.7.4.6 Click next.
10.7.4.7 In the Sample Window, add all detector names with the format: ICB;AV(detector)#.
10.7.4.8 Click next

10.7.4.9 In the Acquisition Window, confirm count time is set at 960 minutes (or as long as the longest sample count time)
10.7.4.10 Click next
10.7.4.11 In the Analysis Set Up Page, select Background Library and Background ROI, check the Use ROI box.
10.7.4.12 Click next
10.7.4.13 In the Report Window, select print on completion
10.7.4.14 Click finish
10.7.4.15 The Detector Assignment worksheet will appear, assign detectors, and select start now.
10.7.4.16 Record the backgrounds in the instrument maintenance log.
10.7.4.17 The background spectrum will be processed by the software
10.7.4.18 The detectors shall be "categorized" after each monthly background. The detectors will be labelled as follows:

Counts in Region of Interest (i.e. Th230, Th232, U234, U238, Pu238, Pu239):

- 0-2 counts – Blue – Ultra Low Level
- 0-4 counts – Yellow – Intermediate Low Level
- 0-20 counts – Green – Low Level
- 0-40 counts –Red – Always designated for Routine analysis when the RL=1 or the activity is from a known radioactive site.

See Attachment 1 Detector Color Key

10.7.4.19 Detectors with backgrounds above the counts listed above are taken out of service for cleaning.
10.7.4.20 Detectors may also be removed from service when there is a visible peak present or at analyst judgment.
10.7.4.21 Backgrounds will be 2nd reviewed before placing into service and a notation of acceptable will be listed in the electronic monthly maintenance log.

10.8 Detector Cleaning (This should be done before starting Backgrounds)
10.8.1 Clean detector surface with ethanol and a clean cotton ball.
10.8.2 Clean the sample tray and place a clean background planchette on the tray.
10.8.2.1 A passing background count is required before returning the detector to service.

10.9 Nuclide Specific Detectors
10.9.1 Due to recoil from thorium isotopes certain ROI may build up background counts affecting isotopes such as Cm-244 and Pu-236. Analyses reporting such isotopes shall not be performed on detectors that count thorium samples.

10.6 Standard Verification Procedure
10.6.2 Receive manual batch from prep
10.6.3 Count for 960 minutes (make sure batch is set up correctly).
10.6.4 After count, open decay corrector (located in Rad Dive, LSC, decay corrector) to see if isotope you are verifying needs to be decay corrected. (if the isotope verifying is located in this spreadsheet, it needs to be decay corrected). Note, new activity on the prep sheet.
10.6.5 Open new spreadsheet verification folder (located in Rad Drive) and select master 3 or 6 point verification (depending on how many standards are made)
10.6.6 Enter calculated value from Decay Corrector (as True value) and value from the spectra print outs (activity on the spectra for the Isotope you are verifying).

10.6.7 Make sure units match.
10.6.8 Standard passes if the mean value is within 5% of certified (true) value, the 1.96 sigma value is within 10% of mean value and the standard reverification acceptablity evaluations are all yes.
10.6.9 Sign bottom of prep sheet and calculation page.
10.6.10 Give to prep supervisior.

11 PROCEDURE

11.6 For sample preparation reference the applicable preparation SOP.

11.7 Initial Setup
11.7.2 Establish the normal instrument settings for all controls.
11.7.2.6 Detector specific high voltage settings and required polarity are listed in the method software settings.
11.7.3 Pulser quality controls shall be checked before each use of the instrument.

11.8 Counting Samples
11.8.2 In Radcapture, go to Utilities > Export>Choose 'Alphavision' or 'Alphavision1' depending on which instrument the samples were set up on>, enter batch # in the window that pops up, and click ok to export to Alphavision.
11.8.3 In Alphavision, go to Process, select Batch to open the Batch Wizard.
11.8.4 Choose Load from LIMS, and pick the batch.
11.8.5 Choose proper analysis by clicking on the correct isotope
11.8.6 Select Next
11.8.7 Click on blank, and then pick blank type (Uu blank, Pu Blank, etc…)
11.8.8 Click on LCS, and then pick LCS type with correct spike number. For amount, use the spike aliquot amount (0.1, 0.2 mL, 0.1326 g, etc) which is printed on the batch paper work.
11.8.9 Select Next
11.8.10 Live time is count time. Enter correct count time for the batch, select next.
11.8.11 Select Nuclide Library, choose correct ROI and the specified tracer that is printed on the batch paper work.
11.8.12 Select next
10.9.2 Select correct activity units (DPM, pCi, etc), select Activity concentration,
10.9.2.1 QuantIMS: change TPU Sigma to 2 (unless otherwise noted in client requirements), add 5% systematic uncertainty and check the negative activity box. .
10.9.2.2 TALS: always select TPU Sigma 1, add 5% systematic uncertainty and check the negative activity box. .
11.8.13 Select Next, two times.
11.8.14 Select Print on Completion.
11.8.15 Select Finish.
11.8.16 Click and drag correct detectors to the correct sample ID and select Start Now.
11.8.17 The spectrum will be processed by the software.
10.9.3 For DOE:
10.9.3.1 FWHM of each tracer peak shall be ≤ 100 keV
10.9.3.2 Tracer peak energy for each sample shall be within ± 50keV of the expected energy.
11.8.18 Backgrounds are checked after high activity samples by counting an 180 minute background with an empty chamber (see 11.9.2).

11.9 Samples with a count rate of greater than 1 CPS should be removed from the alpha counting system to prevent contamination of detector(s).

11.9.2 Alpha detectors exposed to samples with count rates greater than 1 CPS should be tagged out-of-service until an empty chamber check can be performed. To perform an empty chamber check, place a clean stainless steel disc in the chamber, establish vacuum, turn on bias and start acquisition for the pre-set time (180 minutes). Note this in the instrument and maintenance log.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery, RPD, uncertainty, MDC, tracer recovery) and standard instrument software calculations are given in the TestAmerica St. Louis QAM.

12.2 Isotope ROI and libraries are derived from the PCNudat master nuclide library:.

12.2.1 http://www.nndc.bnl.gov/nudat2/indx_dec.jsp

12.3 Any manual integration of a peak or group of peaks must be documented. In all instances where the data system report has been edited or where manual integration has been performed, the operator must clearly identify such edits or manual procedures. Reference SOP ST-QA-0040 for details.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) module. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – If the MB concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – The batch is re-prepped and re-analyzed for the affected isotope.

13.3.2.3 RPD/RER Duplicate excursion – For the RPD/RER one or both must be with in acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (ie, nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narrated in an NCM.

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration

13.5 Sample Result Evaluation

13.5.1 Tracer recovery must be within specified limits. Tracer limits are 30% - 110% unless other wise specified by the client

13.5.2 Tracer/Carrier recovery (low) – Re-extract using a reduced volume or recount for maximum count time to achieve 400 tracer counts

13.5.2.1 Note: QSAS allows for reporting results as quantitative when tracer recoveries are below 30% if:

13.5.2.1.1 the relative uncertainty associated with the tracer recovery is less than 10% (2 sigma)

13.5.2.1.2 spectral resolution requirements are met and there are no indications of spectral interferences (resolution of <100 keV).

13.5.2.1.3 detection limit requirements have been met

13.5.3 Tracer/Carrier recovery (high) – If the blank and LCS are **within limits**, have the sample logged in for native analyisis if not already logged in for native. If the blank and or LCS has **high recovery**, a reprep is required.

13.5.3.1 Truncation to 100%: Truncation can be done at the clients discretion, or with approval from manager or technical director or based on sample history.

13.5.3.2 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.5.3.2.1 minimum number of tracer counts

13.5.3.2.2 level of uncertainty

13.5.3.2.3 client project requirements/approval

13.5.4 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.5.5 The following occurances require a dilution to be performed:

13.5.5.1 Dilution level is determined by taking the highest gross counts divided by the count time divided by a factor of 2. (ie: 7200/180/2=1:20)

$$DL = GCts_{High} / t_{count} / 2$$

$$DL = Dilution\ Level$$

$$GCts_{High} = Highest\ Gross\ Counts$$

$$t_{count} = Count\ Time$$

13.5.5.1.1 Tailing – A peak is significantly tailing out side its region of interest (ROI)

13.5.5.1.2 The tracer recovery is low due the high activity of the sample

13.5.5.1.3 Peak Interference – A Peak is observed which is identified as an interference

13.6 Insufficient Sample

13.6.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis an NCM is written and a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

14.1 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Measurement Laboratory (EML) HASL 300 28th Edition method A-01-R, Alpha Radioassay

17.2 AlphaVision-32, Alpha Particle Spectrum Acquistion and Analysis for Microsoft Windows and NT, Software Version 5.0 Installation, User Interface and Reference Guide, Ortec (latest version)

17.3 OCTETE Plus, Integrated Alpha-Spectroscopy System Hardware Operating Manual, 777720, Ortec (latest version)

17.4 MAESTRO-32, MCA Emulator for Microsoft Windows, A65-B32 Software User's Manual, 777800, Ortec (latest version)

17.5 U.S. Nuclear Regulatory Commission, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment, Regulatory Guide 4.15.

17.6 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

17.7 TestAmerica, St. Louis Quality Assurance Manual, current revision

17.8 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.9 Decay Radiation Datebase, Version of 5/8/2013; http://www.nndc.bnl.gov/nudat2/indx_dec.jsp

17.10 Associated SOPs, current revisions
- 17.10.1 ST-PM-0002, Chain of Custody
- 17.10.2 ST-QA-0002, Standard and Reagent Preparation
- 17.10.3 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.10.4 ST-QA-0036 Non-Conformance Memorandum (NCM) Procedure
- 17.10.5 ST-QA-0040, Manual Integration Procedure
- 17.10.6 ST-RC-0040, Total Alpha Emitting Isotopes of Radium
- 17.10.7 ST-RC-0238, Isotopic Uranium By Eichrom® Uteva Resin For Various Matrices
- 17.10.8 ST-RC-0210, Determination Of Polonium-210 By Alpha Spectrometry
- 17.10.9 ST-RC-0232, Isotopic Thorium And/Or Neptunium In Various Matrices By Eichrom® Teva Separation Resin
- 17.10.10 ST-RC-0240, Isotopic Americium, Curium, Plutionium, Thorium, And Uranium In Various Matrices By Eichrom® Separation Resin
- 17.10.11 ST-RC-0241, Americium, Plutonium, Curium, And Uranium In Various Matrices By Eichrom® Uteva And Tru Resins (With Vacuum Box System)
- 17.10.12 ST-RC-0242, Isotopic Thorium, Plutonium And Uranium In Various Matrices By Eichrom® Separation Resins
- 17.10.13 ST-RC-0246, Isotopic Americium, Curium, Uranium In Various Matrices By Eichrom® Separation Resins

18.0 MODIFICATIONS TO REFERENCE METHOD

18.1 Energy calibrations checks are performed monthly. Daily pulsar checks are performed in place of the weekly energy calibration checks.
18.2 Backgrounds are determined monthly rather weekly
18.3 CCV's are determined monthly rather that before and after each measurement.

19.0 CHANGES TO PREVIOUS REVISION

19.1 No Changes- Annual Review
19.2 Rev. 8:
- 19.2.1 Section 10 additions
  - 19.2.1.1 Addition of Instrument setup as §10.1
  - 19.2.1.2 Addition of Calibration Quality Control Check as §10.3
  - 19.2.1.3 Addition of calibration acceptance criteria

19.3 Rev. 9:
- 19.3.1 Section 10.5, addition of limits for pulser centroid and pulser energy

19.4 Rev. 10:
- 19.4.1 Section 10:
  - 19.4.1.1 2nd level review for ICV and CCV added to section 10
  - 19.4.1.2 1200 minute setting for acquisition window for special projects
  - 19.4.1.3 Upper control limits for long backgrounds
  - 19.4.1.4 Detector cleaning
- 19.4.2 Section 12: addition of ROI and library reference
- 19.4.3 Section 13: Occurances that require dilution
- 19.4.4 Addition of Attachement 1

19.5 Rev. 11: (8/21/13)
- 19.5.1 Grammatical corrections through out and removal of referencest to QuantIMS and Clouseau
- 19.5.2 Section 10, updated internal calibrations information in section and the calibration process in the lab software throughout
- 19.5.3 Section 10, added new standards verification procedure
- 19.5.4 Section 13, added corrective actions and equation for dilution level
- 19.5.5 Section 15, updated with new verbiage
- 19.5.6 Attachment 1 revised
- 19.5.7 Section 13,removed “See Clouseau NCM for Corrective Action”and added specfic Corrective Actions to the SOP
- 19.5.8 Section 18, added backgrounds will be done monthly and added CCV’s will be done monthly.
- 19.5.9 Section 3, Added “keV” definition
- 19.5.10 Section 6, Added Hardware and Software specifics

19.6 Rev. 12: (4/24/15)
- 19.6.1 Section 10.2.1.1, added the energy vs. channel slope should be less than 15 keV per channel. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Changed 5 channels to 2.5 channels and 40 keV to 20 keV.
- 19.6.2 Section 10.2.2.1, added a minimum of 3000 net counts must be collected for each peak in the calibration.
- 19.6.3 Section 10.2.3.1, added a minimum of 2000 net counts must be collected for each peak in the ICV. Added the Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 20 keV of the corresponding calibraation peak in the initial calibration (IC).
- 19.6.4 Section 10.3.1.1, added The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 20 keV of the corresponding calibraation peak in the initial calibration (IC).
- 19.6.5 Added section 10.5
- 19.6.6 Updated calibration identification in procedure

Attachment 1

AlphaVision Detector Key

Red – Routine only 0-40 counts

Green – Low Level 0-20 counts

Yellow – Intermediate Low Level 0 – 4 counts
Special Projects and QC samples (Method Blanks and Lab Control Samples) Only

Blue – Ultra Low Level (for Pu/Am/Np)
Special Projects Only

Out of Service
OOS reason written here

OOS Tag – Out of Service (OOS)

Title: LIQUID SCINTILLATION COUNTER ANALYSIS

Approvals (Signature/Date):

Chris Hough 5/6/13 Chris Hough Date Radiochemistry Manager	Michael Ridenhower 5/6/13 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 5/8/13 Marti Ward Date Quality Assurance Manager	Elaine Wild 5/6/13 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-RD-0302 Rev. 13

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1.0 SCOPE AND APPLICATION

1.1 This procedure provides instruction for the operation of the Packard Tri-Carb series 3170, Packard Tri-Carb series 3180, and Packard Tri-Carb 2550 CA/LL Liquid Scintillation Analyzer.

1.2 This procedure applies to all samples that are analyzed using the liquid scintillation counter, namely:

- Carbon-14
- Iodine-129
- Iron-55
- Lead-210
- Nickel-59 and 63
- Plutonium-241
- Technetium-99
- Tritium
- Total Activity

1.3 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS)..

2.0 SUMMARY OF METHOD

2.1 The liquid scintillation counter is calibrated with standards containing a known quantity of H^3, C-14, Fe-55, I-129, Ni-59/63, Pb-210, Pu-241, and Tc-99. Not every instrument is calibrated for every nuclide. However, any instrument used to analyze a given method is calibrated for the nuclide requested.

2.2 Prepared samples are loaded into the sample tray and counted.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

3.2 Quenching - The interference with the conversion of decay energy to electronic signal in the photo multiplier tubes resulting in a reduction in counting efficiency. This can be caused by materials present in the scintillation solution that interfere with the process leading to the production of light as well as materials present that absorb the light before it reaches the photo multiplier tubes.

4.0 INTERFERENCES

4.1 See individual sample preparation SOPs.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

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5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED
The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Packard Tri-Carb Series 3170, Packard 3180, Packard Tri-Carb 2550 CA/LL Liquid Scintillation Analyzers.

6.2 Liquid Scintillation Counter vials

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI Water Type obtained from the Milli-Q unit.

7.3 Unquenched standards:
- 7.3.1 Background Standard
- 7.3.2 Unquenched C -14 standard
- 7.3.3 Unquenched I-131 standard
- 7.3.4 Unquenched Fe-55 standard
- 7.3.5 Unquenched I-129 standard
- 7.3.6 Unquenched Ni-59/63 standard
- 7.3.7 Unquenched Pb-210 standard
- 7.3.8 Unquenched Pu-241 standard
- 7.3.9 Unquenched Tc-99 standard
- 7.3.10 Unquenched Tc-99 for Total Activity standard
- 7.3.11 Unquenched H3 standard
- 7.3.11 Unquenched Pm-147 standard

7.4 For Quench curves:
- 7.4.1 Quenched C-14 standard
- 7.4.2 Quenched I-131 standard
- 7.4.3 Quenched Fe-55 standard
- 7.4.4 Quenched I-129 standard
- 7.4.5 Quenched Ni-59/63 standard
- 7.4.6 Quenched Pb-210 standard
- 7.4.7 Quenched Pu-241 standard
- 7.4.8 Quenched Tc-99 standard
- 7.4.9 Quenched Tc-99 for Total Activity standard
- 7.4.10 Quenched H3 standard
- 7.4.11 Quenched Pm-147 standard

7.5 Standards may contain varying activities depending on the supplier and concentration availability of the nuclide of interest. However, all standards in a quench curve are counted until a minimum of

10,000 counts are collected.

7.6 Sample to cocktail ratios: See Attachment 1 of this SOP.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 See individual sample preparation SOP for specific sample handling and storage requirements.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), Matrix Spike (MS) and a sample duplicate (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank (DI water) must be prepared with every sample batch.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.4 **Matrix Spike**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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10.0 CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration:

10.1.1 Prepare a quench curve for the nuclide of interest.

10.1.1.1 The process for quench curve preparation can be found on the print out of the previous calibration file.

10.1.2 Frequency:

10.1.2.1 Quench curves are performed minimally on an annual basis.

10.1.2.1.1 A new calibration curve is generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include any significant changes in instrument operating parameters, and major instrument maintenance.

10.1.3 Acceptance Criteria:

10.1.3.1 A minimum of 10,000 counts must be obtained for each data point

10.1.3.2 Correlation Coefficient (r^2) > 0.995 or each data point less than 7.5% relative to the calibrated value calculated from the fitted curve.

10.1.3.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.2 Initial Calibration Verification (ICV)

10.2.1 The initial calibration verification standard is a different standard source than the one used for the initial calibration.

10.2.2 Validate the quench curve by counting at least three second source standards.

10.2.2.1 The process of preparing validation standards is found on the print out of the previous calibration file. The verification standards should contain an unquenched standard as well as quenched standards that represent the quench range of live samples.

10.2.3 Frequency:

10.2.3.1 An ICV is performed with every initial calibration.

10.2.4 Acceptance Criteria:

10.2.4.1 Mean Recovery +/- 10% of the known value.

10.2.4.1.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard.

10.2.4.1.2 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.3 Continuing Calibration:

10.3.1 Load standards and use the appropriate protocol flag as listed on the status page “Daily QC”. The instrument will perform the required tests and the results are printed and saved to the Quality Control Table.

10.3.2 Frequency:

10.3.2.1 Quality control checks are performed for each day of use for the nuclides which measurements are being performed.

10.3.3 Acceptance Criteria:

10.3.3.1 The instruments are set at 2 sigma for a warning and 3 sigma for out of control limits.

10.3.3.2 If a Continuing Check fails and the analyst can document the reason for failure (e.g. carryover from the previous sample, etc.) then a second check may be analyzed without any adjustments to the instrument. If this check meets criteria then sample analysis may continue.

10.3.3.3 If this second check does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require

calibration.

11.0 PROCEDURE

11.1 Confirm the following:
- 11.1.1 Computer software is loaded and operational.
- 11.1.2 Printer is on-line.

11.2 Load sample vials by lifting the instrument cover and placing samples in tray with flag facing towards the left.

11.3 Samples are counted from left to right. QC checks are run daily. Prior to starting samples, make sure the daily QC has run and is acceptable.

11.4 Position 1 in the sample tray is reserved for the background vial.

11.5 Log the following information into the Run Log:
- 11.5.1 Date
- 11.5.2 Batch number
- 11.5.3 Sample I.D.
- 11.5.4 Protocol No.
- 11.5.5 Count length
- 11.5.6 Type of analysis
- 11.5.7 Operator's initials

11.6 Close instrument cover and allow sample vials to dark-adapt per the cocktail manufacturer's instructions, typically 30 minutes.

11.7 Spectrum are assessed for interferences, peak shape, etc. against a known source either the LCS or the instruments reference manual.

11.8 For the Packard 3170 LSC and 3180 LSC:
- 11.8.1 Maximize the instrument window. Double click on the flag icon which has the protocol number that needs to be run. Click on the count conditions tab at the top of the analysis window. Enter the correct count time in the count time field.
- 11.8.2 Click on the Worklist tab at the top of the analysis window. Scan the samples from the Rad Capture paperwork.
- 11.8.3 Verify sample tray is in the correct order, starting with the background, blank, LCS, followed by the samples. Check tray order with the order appearing on the screen version of the worklist.
- 11.8.4 Click on the green start flag in the upper left corner of the instrument window.
- 11.8.5 Upon completion of counting, results will be sent to printer. The data is sent to the appropriate instrument folder.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculations are performed in RadCapture (TestAmerica St. Louis custom software). These calculations are printed directly from the software and are included the TestAmerica St. Louis ST-QAM.

12.2 Commonly used calculations (e.g. % recovery, RPD, uncertainty, MDC, tracer recovery) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR

OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – Blank contamination above the RL (> ½ RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank. An NCM is written to document the excursion.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.3.2.3 RPD/RER Duplicate excursion batch is re-prepped and re-analyzed.

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Recovery excursion may not necessarily warrant corrective action other than narration. If affected analyte activity in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries are out outside limits, sample re-prep and re-analysis are required.

13.5 Sample result evaluation

13.5.1 Tracer recovery must be within specified limits.
Tracer/Carrier recovery low– batch is re-prepped and re-analyzed.

13.5.2 Tracer/Carrier recovery high–.batch is re-prepped and re-analyzed. Samples that are non-detect may be reported with an NCM.

13.5.2.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.5.2.2 Minimum number of tracer counts

13.5.2.3 Level of uncertainty

13.5.2.4 Client project requirements/approval

13.5.2.5 If the sample carrier recovery is significantly higher than normal, the native concentration in the sample of the carrier analyte may be present causing a high bias to the carrier recovery. This high bias to the carrier analyte would in turn

cause a low bias to the samples result. The laboratory defines significant to be an additional 20% above the average LCS/MB carrier recovery (as determined from a population of LCS and MB data), with a maximum of 110%. The table below shows the limits determined for each carrier analyte. The analyst should ensure that the carrier analysis is requested to determine native concentration for samples exceeding the limit.

Iodine	Nickel	Lead
97%	102%	109%

13.5.3 These expectations will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.6 Insufficient Sample

13.6.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report case narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for “Waste Management and Pollution Prevention.”

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator."

17.0 REFERENCES

17.1 EPA-600/4-80-032. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Environmental Monitoring and support Laboratory, Section 10, Method 906, "Tritium in Drinking Water", August, 1980

17.2 Standard Methods for the Examination of Water and Wastewater. 19th Edition, Method 7500-1B Mod "Precipitation Method", 1985

17.3 EPA 520/5-84-006, EERF Radiochemistry Procedures Manual, Method C-01-1, "Radiochemical Determination of Carbon-14 in Aqueous Samples", August 1984

17.4 Eichrom Technologies, Inc. Analytical Procedures, TCS01, "TC-99 in Soil", April, 2002

17.5 Eichrom Technologies, Inc. Analytical Procedures,TCW01, TC-99 in Water, April 2002

17.6 Eichrom Technologies, Inc. Analytical Procedures,OTW01, Pb-210 in Water, April 2001

17.7 Eichrom Technologies, Inc. Analytical Procedures,OTS01, Pb-210 in Soil, August, 2002

17.8 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.10 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

17.11 Associated SOPs, current revisions;

17.11.1 ST-PM-0002, Sample Receipt and Custody
17.11.2 ST-QA-0002, Standard and Reagent Preparation
17.11.3 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.11.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.11.5 ST-RC-0030, The Determination of Tritium in Water (and Other Fluids), Soil and Silica Gels
17.11.6 ST-RC-0031, Tritium Determination by Cryogenic Distillation
17.11.7 ST-RC-0042, Iodine-129 in Water
17.11.8 ST-RC-0055, Determination of Iron-55, Nickel-59 and Nickel-63 by Liquid Scintillation Spectrometry
17.11.9 ST-RC-0056 , Carbon-14 by Liquid Scintillation Spectrometry
17.11.10 ST-RC-0125, Determination of Technetium-99 using Eichrom® TEVA Resin
17.11.11 ST-RC-0211, Determination of Lead-210 by Liquid Scintillation Counting
17.11.12 ST-RC-0245 , The Determination of Pu-241 by Liquid Scintillation Counting
17.11.12 ST-RC-0247, Promethium-147 and Samarium-151 analysis Utilizing Lanthide Resin Separation Procedure

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 EPA method 906 is a drinking water method, and as such formulated its' Tritium calculation to reflect counts per minute (CPM) rather than disintegrations per minute (dpm). In true drinking water samples, it is acceptable to use CPM since drinking water samples should have little effect on efficiencies. However, EPA method 906 is commonly referenced for wastewater samples, and even silica gels and soil matrices. For these matrices, there can be a significant difference in efficiency and thus using dpm provides a more accurate result.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Added Promethium back to the list of standards in section 7.3.11 and 7.3.10.

19.2 Rev 13:

- 19.2.1 Added Iodine-131 and Total Activity to section 1.2.
- 19.2.2 Added Iodine-131 to section 2.1.
- 19.2.3 Added section 7.6 and attachment 1 to document sample to cocktail ratios
- 19.2.4 Removed redundant section 18.2

19.3 Rev 14:

- 19.3.1 Replaced TALS and Clouseau references with LIMS
- 19.3.2 Removed Packard 2550 instructions from section 11
- 19.3.3 Added native carrier to section 13
- 19.3.4 Removed Clouseau NCM in Section 13 and added specific corrective actions.

Attachment 1

LIQUID SCINT. SAMPLE TO COCKTAIL RATIOS

ST-RC-0015	Total Activity		
Solid and Semi-solid Samples			
	0.25 - 3	mL	Sample
	15 - 17	mL	Cocktail
	18	mL	Final
Aqueous Samples			
	0.25 - 3	mL	Sample
	15 - 17	mL	Cocktail
	18	mL	Final
Oil Samples			
	3	mL	Sample
	15	mL	Cocktail
	18	mL	Final

ST-RC-0030	Tritium		
Steam Distillation (Water); Silica Gel; Solids (soil, filters/swipes); Direct Method - Water			
	10	mL	Sample
	10	mL	Cocktail
	20	mL	Final
Direct Method - Oil			
	1 - 2	mL	Sample
	18 - 19	mL	Cocktail
	20	mL	Final

ST-RC-0031	Tritium Cryo		
	10	mL	Sample must dilute to 10 mL total if 10 mL of sample not present
	10	mL	Cocktail
	20	mL	Final

ST-RC-0042	I-129		
Carrier Determination by ICP-MS			
	4.9	mL	Sample
	15	mL	Cocktail
	19.9	mL	Final
gravimetric carrier			
	5	mL	Sample
	15	mL	Cocktail
	20	mL	Final

ST-RC-0055	I-55 & Ni-59/63		
Iron			
	4.9	mL	Sample
	15	mL	Cocktail
	19.9	mL	Final
Nickel			
	3.9	mL	Sample
	16	mL	Cocktail
	19.9	mL	Final

ST-RD-0302_LqScint-Sample2CocktailRatio.xls Rev (0)

LIQUID SCINT. SAMPLE TO COCKTAIL RATIOS

ST-RC-0056	**C-14**		
	10 mL	Sample	
	10 mL	Cocktail	
	20 mL	Final	
ST-RC-0125	**Tc-99**		
	2 mL	TEVA resin	
	5 mL	dI Water	
	10 mL	Cocktail	
	17 mL	Final	
ST-RC-0211	**Pb-210**		
	5 mL	Sample	
	15 mL	Cocktail	
	20 mL	Final	
ST-RC-0245	**Pu-241**		
	4 mL	Sample	0.1 mL dI water rinse used
	16 mL	Cocktail	
	20 mL	Final	
ST-RC-0247	**Pm-147**		
	10 mL	Sample	
	10 mL	Cocktail	
	20 mL	Final	

ST-RD-0302_LqScint-Sample2CocktailRatio.xls Rev (0)

Title: LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING (GFPC) SYSTEM ANALYSIS

Approvals (Signature/Date):

Chris Hough 1/16/15 Chris Hough Date Radiochemistry Department Manager	Michael Ridenhower 1/16/15 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 1-16-15 Marti Ward Date Quality Assurance Manager	Elaine Wild 1/16/15 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-RD-0403 Rev. 14

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to all Low Background Proportional Counting instruments. TestAmerica St. Louis performs radium-226/228, strontium-89/90, gross alpha/beta, neptunium-36 and chlorine 36.

1.2 This SOP is based on SW846 method 9310, 9315 and 9320; EPA methods 900.0, 903.0, 904.0, 905.0; and DOE EML HASL 300 method, Ba-01-R, Sr-02 and Sr-03-RC.

1.3 The SOP applies to GFPC analysis of liquid and solid matrices.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This procedure provides instructions for the daily calibration and maintenance of the Low Background Proportional Counting instrumentation.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualifiers.

3.2 IQC - a computerized Quality Control Program where the counting results of Daily Radioactive check sources and Daily Background checks are entered and compared to statistical average data. A measurement within ± 3 standard deviations indicates the detector is operating within acceptable parameters.

3.3 αLL - discriminator setting indicating the alpha lower voltage limit.

3.4 Alpha Voltage Only - detector voltage capable of collecting ions created by alpha radiation only. Ion pairs created by beta radiation are not collected.

3.5 αUL - discriminator setting indicating the instruments alpha upper voltage limit.

3.6 βLL - discriminator setting indicating the beta lower voltage limit.

3.7 βUL - discriminator setting indicating the beta upper voltage limit.

3.8 Crosstalk - a measure of the amount of beta radiation that is collected in the alpha radiation channel; it is also a measure of alpha radiation collected in the beta channel.

3.9 Plateau - a point on a graph of count rate vs. detector bias voltage where further increases in bias will not result in an increase in measured counting rate.

3.10 LB4100 – LBPC (Low background Gas Flow Proportional Counting instrument).

4.0 INTERFERENCES

4.1 A detector contaminated with radioactive material will result in a high background and interfere with the correct measurement of a sample.

4.1.1 If a sample “times out” reaching 10,000 counts before the allotted time, and the sample count rate is 60 cpm or greater, then another daily background check is performed on that detector. If the detector background check is unacceptable, the detector is taken Out Of Service until action is taken to bring the background check within acceptable limits. If the chamber requires action to remove contamination and a new background check is acceptable, then a 30 minute empty chamber count should be performed to determine if a new long background needs to be performed on that detector.

4.2 The actual counting efficiency for alpha radiation decreases greatly with a density > 6.0 mg/cm2. Therefore, the maximum acceptable mass density is typically 5 mg/cm2 or less that 100 mg for a 2" planchet.

4.3 For beta radiation, reliable data may be obtained counting samples with a density as high as 10 mg/cm2 or greater.

4.4 Sample thickness as well as moisture content may impact the alpha and/or beta results.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Silver Nitrate	Poison Corrosive Oxidizer	$0.01^{g}/_{m3}$ (TWA) for silver, metal dust, and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Skin contact may cause redness, pain, and severe burning. Eye contact can cause blurred vision, redness, and pain.
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH_3)	Inhalation symptoms may include irritation to the respiratory tract. Ingestion symptoms may include pain in the mouth, chest, and abdomen with coughing, vomiting, and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 Low Background Proportional Counter, equivalent to the Canberra/Oxford/Tennelec LB4100, or Protean MPC9604.

6.2 Gas mixture, 90% argon, 10% Methane
6.3 Blank planchets
6.4 PC based data acquisition system, IQC software
6.5 Centrifuge tubes
6.6 Centrifuge
6.7 Vortex
6.8 Pipettes, Eppendorf or equivalent
6.9 Pipette, disposable

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision

7.2 Radioactive sources to measure beta radiation,: Sr-90 and Ra-228 sources.

7.3 Radioactive sources to measure alpha radiation: Am-241, Th-230 and Ra-226

7.4 Deionized Water (DI), obtained from the Milli-Q unit.

7.5 Silver nitrate ($AgNO_3$), 0.5 N

7.6 Sodium chloride (NaCl), crystals

7.7 Sodium chloride (NaCl), 0.5 N

7.7.1 Add 50 mL of DI water to a 100 mL volumetric, add 5.84 g of NaCl, dilute to 100 mL, cap and shake to dissolve. Adjust volume to 100 mL with DI water.

7.8 Ammonium hydroxide (NH_4OH), concentrated, 28 N

7.9 Ammonium hydroxide (NH_4OH), 5 %

7.9.1 Add 25 mL of concentrated Ammonium Hydroxide to 475 mL of DI water. **CAUTION – Ammonium hydroxide is corrosive. Mist and vapor cause burns to every area of contact.**

7.10 Cl-36: At least four sodium chloride standards are prepared for calibration.

7.10.1 Add 10 mL of DI water to 4 centrifuge tubes.
7.10.2 Add 0.500 mL of 0.5 N sodium chloride carrier solutions to each centrifuge tube. Swirl to mix.
7.10.3 Add 2 drops of 5 % ammonium hydroxide solution, swirl to mix.
7.10.4 Add 12 mL of 0.5 N silver nitrate solution to each centrifuge tube.
7.10.5 Vortex for 30 seconds.
7.10.6 Centrifuge and decant supernate to waste.
7.10.7 Proceed to section 11.4, Planchet Preparation of Silver Chloride Precipitation of SOP ST-RC-0036.
7.10.8 Average the four weights for the sodium chloride carrier solution, record the standardized weight in the log book and on the bottle.
7.10.9 NOTE: It may be necessary to use more than 0.500 mL of carrier in some large water samples or calibrate a 4 N sodium chloride carrier solution. The efficiency of the detectors will have to be calculated using the heavier sodium chloride carrier solution.

7.10.10 Prepare four sodium chloride calibration samples as in Section 7.10 but add a known amount of Cl-36 to each tube before the sodium chloride carrier is added. Analyze samples by GFPC and determine detector efficiency as per Section 12, Data Analysis and Calculations.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 See associated sample preparation SOPs ST-RC-0020, ST-RC -0021, ST-RC -0036, ST-RC -0040, ST-RC -0041and ST-RC -0050, for more detailed information.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank (MB)**

9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 A method blank must be prepared with every sample batch.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 An LCS is a blank matrix spiked with a known amount of target analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 An LCS must be prepared with every sample batch.

9.5 **Matrix Spike**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate**

9.6.1 A Sample Duplicate is an additional aliquot of a field sample, processed simultaneously with, and under the same conditions as, samples through all steps of the analytical process to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**

9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Additional preventative maintenance can be found in ST-QA-0024.

10.2 Voltage Plateau Determination

10.2.1 **Frequency**:

10.2.1.1 Performed as a part of the Intial Calibration.

10.2.2 **Voltage Plateau Determination on Protean MPC 9604**

10.2.2.1 Place the Sr 90 source or sources in the detector drawer.

10.2.2.2 Select detector of interest on the computer screen.

10.2.2.3 Click Plateau under Count Method.

10.2.2.4 Set time to 5min for distributed Sr90 source.

10.2.2.5 Select A, B. C, D.

10.2.2.6 Click OK.

10.2.2.7 When count is complete select Plateau under instrument Specific.

10.2.2.8 Set Beta appropriate voltage with arrows </>. Evalulate and Print report.

10.2.3 **Criteria for Plateaus for Protean MPC 9604**

10.2.3.1 Acquire 40 data points in 30V increments beginning at 705V and ending at 1875V. Slope should be no more than 5%.

10.3 **Discriminator Settings**

10.3.1 **Frequency:**

10.3.1.1 Performed as a part of the Intial Calibration.

10.3.2 **Discrimator Settings on Protean MPC 9604**

10.3.2.1 Collect a minimum of 10,000 counts for each of Am-241, Th-230 and/or Po-210 sources

10.3.2.2 Calculate the percentage of crosstalk and compare the results to historical and expected values. Consult the Technical director if the values fall out of range.

10.4 **Initial Calibration (IC)**:

10.4.1 **Frequency**:

10.4.1.1 The Gas Flow Proportional Counter (GFPC) is calibrated initially and verified each year thereafter. Recalibration may be required if indicated during the operation of the instrument.

10.4.2 The specific calibration source preparations can be found in the file containing the previous calibration.

10.4.3 All nuclide sources shall be NIST traceable.

10.4.4 The efficiency calibration shall consist of at least seven mass attenuated calibration standards, unless a single point source efficiency is to be determined.

10.4.5 Alpha, Beta Ra226 - at least seven mass attenuated calibration standards

10.4.6 Air Filter – single point calibration

10.4.7 Cl-36 – Averaged 4 point calibration.
10.4.8 Np – Averaged 4 point calibration.
10.4.9 The standards shall have enough activity to generate at least 10,000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5,000 counts per second.
10.4.9.1 For alpha and beta analysis, separate sets of calibration sources shall be prepared.
10.4.10 The mass attenuation is accomplished by utilization of a salt solution with comparable make up to the majority of samples seen in the laboratory.
10.4.10.1 Alternatively, the mass attenuation may be accomplished by using the same carrier solution used in a specific analysis.
10.4.11 Each standard shall be counted in every detector to be calibrated.

10.4.12 **IC Criteria:**
10.4.12.1 The efficiency of the detector (the dependent variable) shall be plotted on a single graph against the masses (the independent variable) for all data points.
10.4.12.2 The equation of the calibration curve shall be determined using polynomial functions and be included on the plot of the curve. The curve shall be continuous and smooth.
10.4.12.3 The degree of the polynomial shall not exceed three. The number of discreet source pairs shall be two more than the degree of the polynomial.
10.4.12.4 The percent difference of the measured efficiency and theoretical efficiency shall be calculated for all data points.
10.4.12.5 Points that are visual outliers or demonstrate less than 15 percent difference between the measured efficiency and theoretical efficiency may be removed at the analyst's discretion. Low residual mass sources and samples are difficult to plate with acceptable duplicate precision. Therefore, high outliers may not necessarily be removed from the calibration if they mimic live sample masses. In any case outliers above 15 percent shall be removed from the calibration curve. No more than 20 percent of the data points may be removed. Reasons for removal or inclusion of outliers shall be documented in the calibration narrative. Once outliers are removed, the percent difference between the measured efficiency and theoretical efficiency must be recalculated using the new polynomial coefficients generated from removal of data points. If outliers over 15 percent difference remain between the measured efficiency and theoretical efficiency the Radiochemistry Manager/QA must be consulted before calibration may continue.
10.4.12.6 The coefficient of determination (r^2) shall be calculated and displayed on the plot with the equation of the trend line. An r^2 greater than or equal to 0.9 is required to proceed to counting of verification sources.

10.5 **Independent Calibration Verification (ICV)**
10.5.1 **Frequency**:
10.5.1.1 Performed with every intial calibration
10.5.2 GFPC initial calibrations must be verified by a second source standard.
10.5.3 The ICV standard is NIST traceable.
10.5.4 The ICV is counted to accumulate at least 5,000 counts.

Annual Calibration Verification (ACV)

10.5.5 **Frequency**:

10.5.5.1 Performed annually after every initial calibration

10.5.6 GFPC annual calibrations must be verified by a second source standard.

10.5.7 The ACV standard is NIST traceable.

10.5.8 The ACV is counted to accumulate at least 5,000 counts.

10.5.9 **ICV / ACV Criteria**:

10.5.9.1 Prepare 3 verification sources varying in expected mass (low, medium and high) within the calibration range of the curve, unless a single point source is to be determined.

10.5.9.2 Alpha and Beta 3 sources

10.5.9.3 Ra226 single source

10.5.9.4 Air Filter single source

10.5.9.5 Cl-36 – single source

10.5.9.6 Np- single source

10.5.9.7 The source/standard used for the ICV shall be from an independent second source as defined within the laboratory Quality Assurance Manual."

10.5.9.7.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.

10.5.9.8 Count the secondary source in all detectors that were calibrated.

10.5.9.9 Calculate the results in terms of percentage recovery.

10.5.9.10 Calculate the mean results of all masses across each detector.

10.5.9.11 **Criteria:**

10.5.9.11.1 Individual points are within 30 percent of the true value

10.5.9.11.2 The mean result of all masses across all detectors is less than 10 percent.

10.5.9.11.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.6 **Setting Performance Check Criteria After Calibration**

10.6.1 Twenty background check samples are counted and used to establish quality control limits for the daily background checks.

10.6.2 The limits for the background check sample will be established with five points from four months. Every month the oldest months points will be removed and points from the current month will be added.

10.6.3 Twenty alpha/beta check sources are counted after calibration and used to establish quality control limits for the daily source checks.

10.6.4 The limits for alpha/beta check sources will be a running average of the four months post calibration.

10.6.4.1 The limits are to be documented.

10.6.4.2 The limits will be re-established monthly at the following frequency

10.6.4.2.1.1 1st month – take first five data points from the new month and fifteen data points from the initial calibration.

10.6.4.2.1.2 2nd month – take first five points from new month, five from prior month and ten from initial calibration.

10.6.4.2.1.3 3rd month – take first five points from new month, five points each from the previous two months and five from the initial calibration.

10.6.4.2.1.4 4th month – take first five data points from new month and five points each from the previous three months.

10.6.4.3 Limits are set.

10.7 **Alpha to Beta Crosstalk Determination**

10.7.1 The mean mass is determined for each data point used to calculate the mass attenuation curve.

10.7.1.1 These curves are calculated and plotted and the percent of alpha into beta crosstalk is determined. This is done by dividing the beta counts per minute as observed in the beta channel from the alpha calibration source counts by the sum of the alpha and beta counts per minute.

10.7.1.2 The mean percent of alpha into beta is determined for each mass point by using the count data accumulated for two sets of alpha sources.

10.7.1.3 The crosstalk curve is plotted as mean crosstalk values relative to the mean mass for the two sets of data.

10.7.1.3.1.1 In this manner the crosstalk factor can be determined for any given mass.

10.7.1.4 The equation of the curve shall be determined using polynomial functions.

10.7.1.5 The coefficient of determination (R^2) shall be calculated and displayed on the plot as well as the equation for the trendline.

10.8 **Beta to Alpha Crosstalk Determination**

10.8.1 Since beta to alpha crosstalk does not vary across mass, a mean beta to alpha crosstalk correction factor is calculated.

10.8.2 The percent of beta into alpha is determined by dividing the alpha counts per minute as observed in the alpha channel from the beta calibration source counts by the sum of the alpha and beta counts per minute.

10.8.3 The mean percent of beta into alpha is determined for all mass points. The mean percent is insignificant in calculating results, therefore is not applied to the result calculation.

10.9 **Long Background**

10.9.1 **<u>Frequency</u>**:

10.9.1.1 Monthly or whenever instrument conditions have significantly changed since the previous background was performed (e.g. detector replaced, etc.)

10.9.1.2 Minimum count time: 1000 minutes.

10.9.2 Wash the planchet holder and clean the drawers with a 20% radiac wash or ethyl alcohol.

10.9.2.1 <u>Do not spray cleaner directly onto the drawers. Spray cleaner on a Kimwipe, a cotton ball, or paper towel and wipe out the drawers.</u>

10.9.3 Check that instrument settings are as specified in 11.1.

10.9.4 **<u>Protean Long Background Count Set Up</u>**

10.9.4.1 Create Manual batch in RadCapture

10.9.4.2 Export Manual batch from RadCapture

10.9.4.3 At Protean instrument:

10.9.4.4 Select 'Detector'
10.9.4.5 Select 'Sample Log'
10.9.4.6 Select appropriate Long Background (ex: ICB;00) you want to start under sample ID
10.9.4.7 Change count time to 1000min
10.9.4.8 Select 'Start'
10.9.4.9 Continue these steps with detectors 1-15. .
10.9.4.10 Review the data for acceptance when the backgrounds are complete.

10.9.5 **Printing Protean Long Backgrounds**

10.9.5.1 Select 'Print Protean data' icon on the desk top
10.9.5.2 Select OK
10.9.5.3 Enter Batch #
10.9.5.4 Print

10.9.6 **Protean Long Background Entry into Protean**

10.9.6.1 Select Input data
10.9.6.2 Select Definitions
10.9.6.3 Select Calibrations
10.9.6.4 Select Properties
10.9.6.5 Select References 0-7 for Detectors 0 thru 7 and 8-15 for Detectors 8 thru 15
10.9.6.6 Enter Background CPM's for Alpha and Beta from printed data sheet

10.9.7 **Orange and Purple Long Background Count Set-Up**

10.9.7.1 Select detector 0
10.9.7.2 Select 'source log'
10.9.7.3 Select 'ICB' by clicking on the file list arrow.
10.9.7.4 Ensure count time is set to 1000 minutes.
10.9.7.5 Select 'start'
10.9.7.6 Continue these steps with detectors 1-23.
10.9.7.7 Review the data for acceptance when the backgrounds are complete.

10.9.8 **Printing Orange and Purple Long Backgrounds**

10.9.8.1 Select 'Data
10.9.8.2 Select 'Source Count Data'
10.9.8.3 Select 'Source Name' ICB
10.9.8.4 Select 'This Range' enter your date range that Long Backgrounds were performed.
10.9.8.5 Select 'Refresh'
10.9.8.6 Select 'Source Count Summary' under Reports
10.9.8.7 Select 'Print'
10.9.8.8 Select 'Landscape' under Orientation
10.9.8.9 Select 'OK'

10.9.9 **Long Background Criteria:**

10.9.9.1 Long backgrounds are evaluated at ± 3 sigma.
10.9.9.2 Protean is evaluated at < 0.2 CPM for alpha and < 2.0 CPM for beta, due to the lack of capability to control chart for sigma evaluation.
10.9.9.3 The data report is evaluated per detector.
10.9.9.4 If a detector is above this limit, discard planchet.

10.9.9.5 Clean the planchet holder with radiac wash, ethyl alcohol or a detergent spray cleaner and dry thoroughly.
10.9.9.6Place a clean planchet in the holder and repeat steps for that detector (s) only.
10.9.9.7 Perform a new background.
10.9.9.8 Note: The detector is tagged with an out of service tag noted with LMB and date. Detector is out of service until a successful background has been achieved.

11.0 PROCEDURES

11.1 Initial Setup

11.1.1 Check the normal instrument settings for all controls as described below:

11.1.1.1 Tank Flow 8 psi

11.1.1.2 Flow Cells >/= 0.3 SCFH, the flow will vary, the target range is 0.15 to 0.20 SCFH.

11.1.2 The High Voltage is set as indicated in the Manuals for the LB4000/LB4100 located in the count room file cabinet. The Protean remains as set by the manufacturer and does not require adjustment.

11.1.3 If counting gas has just been changed or turned on, allow a minimum purge time of 30 minutes prior to operation. Record gas tank changes on document on separate sheet.

11.2 Record date of Daily Background and Check Source Data in runlog logbook.

11.3 Maintenance

11.3.1 Change out the counting gas when the gauge reads under 500 psi. This usually occurs every 1 to 2 weeks. Record in the instrument maintanence logbook.

11.3.2 Allow gas to purge a minimum of 30 minutes prior to operation.

11.4 Data Acquisition: Daily Background Check and Source Check

11.4.1 **Daily Background Check**:

11.4.2 **Protean Instrument**:

11.4.2.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.
11.4.2.2 Double click detector 0 on the Protean computer screen.
11.4.2.3 Select ‘source log’
11.4.2.4 Set the time for 200 minutes.
11.4.2.5 Type or scan ‘CCB;00’ in the sample id box. (B is for background and 0 is for detector.)
11.4.2.6 Select ‘start’
11.4.2.7 Double click detector 1 on the computer screen. Repeat steps 11.4.2.3 through 11.4.2.5 for each detector, making sure to change the number to coincide with the detector the background is counting for.
11.4.2.8 Remove planchets from detector drawers when counting is complete.
11.4.2.9 On any work station, i.e. “PC computer in the count room”, double click on the IQC icon.
11.4.2.10 Select ‘import data’
11.4.2.11 Select ‘Protean’. Enter the current date. Click on the file list arrow.
11.4.2.12 Select ‘close’
11.4.2.13 Select ‘reporting’. Verfiy the current date in both the ‘start’ and ‘end’ date fields. Select ‘print’ to generate the report.

11.4.3 **Orange and Purple Instrument**:

11.4.3.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.
11.4.3.2 Select detector 0.
11.4.3.3 Select 'source log'.
11.4.3.4 Select or scan 'CCB' by clicking on the file list arrows for orange.
11.4.3.5 Select 'CCB' by clicking on the file list arrows for purple.
11.4.3.6 Select 'start'
11.4.3.7 Repeat these steps with detectors 1-23.

11.4.4 **Daily Background Criteria:**

11.4.4.1 Review the IQC report for each detector.
11.4.4.1.1 If a detector fails background criteria (3 sigma), clean the detector with radiac wash or ethyl alcohol and re-count.
11.4.4.1.2 Tag detector out of service with a Tag noted with "Bkg RC".
11.4.4.1.3 ,If detector fails Background re-count tag detector with an out of service tag noted with date to indicate that the detector is out of service for the day.

11.5 **Daily Source Check**

11.5.1 **Protean Instrument:**

11.5.1.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15 and slowly insert each drawer into the instrument.
11.5.1.2 Double click detector 0 on the Protean computer screen.
11.5.1.3 Select 'source log'.
11.5.1.4 Set the time for 2 minutes.
11.5.1.5 Type or scan "CCVA-"#";SA00" in the sample id box. ("#" is the source container, S is for source, A is for Alpha and 0 is the detector.)
11.5.1.6 Select 'start'
11.5.1.7 Double click detector 1 on the computer screen. Repeat steps 11.5.2.3 to 11.5.2.6 for each detector.
11.5.1.8 When the counting is complete, slowly open each detector drawer. Place beta sources in detectors 0-7. Place alpha sources in detectors 8-15.
11.5.1.9 Double click detector 0 on the Protean computer screen.
11.5.1.10 Type or scan "CCVB-"#";SB00" in the sample ID box. ("#" is the source container, S is for source, B is for Beta and 0 is the detector.)

11.5.2 Double click detector 1 on the computer screen. Repeat steps 11.5.2.10 for each detector.

11.5.2.1 Remove sources from detector drawers when counting is complete
11.5.2.2 Review the IQC report for each detecctor.
11.5.2.3 Limits are +/- 3% (fail).
11.5.2.4 If detector fails source check a Red tag is placed on the outside of the detector to indicate detector is out of service for the day.

11.5.3 **Orange and Purple Instrument**:

11.5.3.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15. Slowly insert each drawer into the instrument.
11.5.3.2 Select detector 0.

11.5.3.3 Select 'source log'.
11.5.3.4 Select 'CCVA-"#";SA00.
11.5.3.5 Select 'start'
11.5.3.6 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15 select 'CCVA-"#";SA08', CCVA-"#";SA09'', and so on for each correlating detector number.
11.5.3.7 Slowly open each detector drawer when counting is complete. Place beta sources in detectors 0-7 and place alpha sources in detectors 8-15.
11.5.3.8 Select detector 0.
11.5.3.9 Select 'CCVB-"#";SB00'.
11.5.3.10 Select 'start'.
11.5.3.11 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15, select 'CCVB-"#";SB08', 'CCVB-"#";SB09' and so on for each correlating detector number.
11.5.3.12 Repeat steps 11.5.4.1 to 11.5.4.11 for detectors 16-23.
11.5.3.13 Remove sources from detector drawers when counting is complete.
11.5.3.14 Review the IQC report for each detecctor.
11.5.3.15 Limits +-3% (fail)
11.5.3.15.1 The indiviual loading samples will verify that detectors are in service.

11.5.4 **Daily Source Criteria:**
11.5.4.1 Review and save with your name and date on the IQC report for each detector.
11.5.4.1.1 If a detector fails criteria, re-count source.
11.5.4.1.2 If detector fails source re-count tag detector with a Red out of service tag noted with date to indicate that the detector is out of service for the day

11.5.5 **Daily check Criteria:**
11.5.5.1 Review and save with your name and date on the IQC report.
11.5.5.1.1 The indiviuals loading samples will verify that detectors are in-service prior to loading on them.
11.5.5.1.2 In addtioin Daily checks will be verified at 1st level review of Data.
11.5.5.1.3

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 Result calculations are performed by TestAmerica St. Louis' Rad Capture software program. These calculations are found in the TestAmerica St. Louis ST-QAM.

12.3 To calculate the efficiency of the detectors for Cl-36, divide the net counts determined of the spiked Sodium Chloride, by the known dpm of the Standard used.

$$\frac{Net\,Counts\,of\,Spiked\,Silver\,Chloride}{Known\,dpm\,of\,Cl-36\,(decay\,corrected\,to\,day\,counted)} = Efficiency$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – (e.g. reprep/reanalysis, narration). If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed..

13.3.2.2 LCS Spike Recovery excursion (low) the batch is re-prepped and re-analyzed for the affected isotope.

13.4 RPD/RER Duplicate excursion – For the RPD/RER One or both must be with in acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (ie, nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narated in an NCM.

13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.5.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample Result Evaluation

13.6.1 Tracer/Carrier recovery must be within specified limits.

13.6.2 Tracer/Carrier recovery low– Samples must be reextracted. Exceptions can be made and results reported with approval from the technical director, manager, or client and approvpriate NCM included.

13.6.3 Tracer/Carrier recovery high

13.6.3.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.6.3.1.1 minimum number of tracer counts

13.6.3.1.2 level of uncertainty
13.6.3.1.3 client project requirements/approval

13.6.4 If the sample carrier recovery is significantly higher than normal, the native concentration in the sample of the carrier analyte may be present causing a high bias to the carrier recovery. This high bias to the carrier analyte would in turn cause a low bias to the samples result. The laboratory defines significant to be an additional 20% above the average LCS/MB carrier recovery (as determined from a population of LCS and MB data), with a maximum of 110%. The table below shows the limits determined for each carrier analyte. The analyst should ensure that the carrier analysis is requested to determine native concentration for samples exceeding the limit.

Radium	Strontium	Chloride
110%	109%	109%

13.6.5 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.7 Insufficient Sample

13.7.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13

of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 ANSI N42.25-1997 – American National Standard Calibration and Usage of Alpha/Beta Proportional Counters

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Ba-01-R, Beta Radioassay, Sr-02 Strontium 90, Sr-03-RC Strontium-90 in Environmental Samples.

17.3 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 1, Method 900.0 Gross Alpha and Gross Beta Radiochemistry

17.4 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0 Alpha-Emitting Radium Isotopes

17.5 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0 Radium-228

17.6 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water

17.7 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9310, Gross Alpha and Gross Beta

17.8 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9315, Alpha-Emitting Radium Isotopes

17.9 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9320, Radium-228

17.10 TestAmerica St. Louis Quality Assurance Manual, current revision

17.11 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions

17.12 Associated SOPs, current revisions:

17.12.1 ST-PM-0002 "Sample Receipt and Chain of Custody"

17.12.2 ST-QA-0002, "Standards and Reagent Preparation."

17.12.3 ST-QA-0024, "Preventative Maintenance"

17.12.4 ST-QA-0036, "Non-Conformance Memorandum (NCM) Process"

17.12.5 ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis".

17.12.6 ST-RC-0020, "Determination of Gross Alpha/Beta Activity"

17.12.7 ST-RC-0021, "Gross Alpha Radition in Water using Copreciptation"

17.12.8 ST-RC-0036, "Determination of Chlorine-36 in Various Matrices by GFPC"

17.12.9 ST-RC-0040, 'Total Alpha Emitting Isotopes of Radium"

17.12.10 ST-RC-0041, "Radium 228 in Water"

17.12.11 ST-RC-0050, "Preparation of Strontium-89 and 90"

17.12.12 ST-RC-0300, "New Jersey 48-hour Gross Alpha Testing for Private Well Testing ACT (PWTA)

18.0 MODIFICATIONS TO THE REFERENCE METHOD

18.1 TestAmerica St. Louis uses thorium-230 to calibrate the GFPC system for Ra-226. Th-230 has similar alpha energies and a sufficiently long half life to eliminate the need for purification. The laboratory has historically performed well on PE programs for Ra-226, demonstrating the laboratory's ability to accurately calibrate for this isotope. Calibrating with a Ra-226 source presents a severe bias in the quantitated result. Ra-226 can be purified and separated from all other alpha emitting isotopes, but the moment after separation, alpha emitting daughters begin to grow (i.e. radon-222, polonium-28 and polonium-214). As the daughter's in-growth alpha activity changes and due to the higher alpha energies of these daughters, the measured efficiency of the GFPC changes as well. After three weeks the alpha activity from purified Ra-226 increases by a factor of four. Due to their short half lives, these daughters can not be isolated long enough to mathematically correct for the bias brought on by them. Calibrating the GFPC with Ra-226 is actually calibrating with a mix of the four isotopes and not a legitimate calibration under the cited regulation.

18.2 Strontium-89 short half life makes it impractical to use as a calibration standard for both radium-228 analysis, as stated in EPA method 904 and SW method 9310, and strontium-89 analysis, as stated in EPA method 905. TestAmerica St. Louis uses a mixed strontium-90/yittrium-90 standard for its' GFPC beta calibration used in Gross Beta, strontium-90, strontium-89, and radium-228 analyses. TestAmerica St. Louis has selected the strontium-90/yittrium-90 standard because it produces a stable beta emission which can be reliably used for initial and continuing calibration. By using this standard mix, we have beta emissions at the lower and upper energetic spectrum whose average is in the middle of the beta range.

18.3 For Ra-228 analysis, TestAmerica St. Louis uses chemical separation techniques to eliminate other potential beta emitters.

18.4 TestAmerica St. Louis does not perform a direct strontium-89 analysis. TestAmerica St. Louis provides calculated results based on the difference between Total strontium and strontium-90.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated Section 10 to address voltage increase per step, plateau slope and QC check count requirements (5000 counts)

19.2 Rev. 11;
- 19.2.1 Added instument Purple throughout section 10 and 11.
- 19.2.2 Adjusted procedure steps throughout section 11.

19.3 Rev. 12,
- 19.3.1 Added Sr-02-RC and Sr-03-RC to sections 1.0 and 17.0.

19.4 Rev. 13:
- 19.4.1 Added Neptunium to scope in section 1.0.
- 19.4.2 Updated the Quality Control Program for counting daily rad checks and daily background checks in section 3.0.
- 19.4.3 Updated background count set-up, printing and entering protean data in section 10.8.

19.5 Rev. 14: (9/12/2013)
- 19.5.1 Removed references to Clouseau, SAC and QuantIMS
- 19.5.2 Section 5.0 added silver nitrate and ammonium hydroxide
- 19.5.3 Section 6.0 updated to include additional equipment
- 19.5.4 Section 7.0 updated to include addition reagents
- 19.5.5 Section 9.0 added reference to prep SOPs for additional information
- 19.5.6 Section 10.0 added sodium cloride standard preparation & reference to ST-QA-0024
- 19.5.7 Section 12.0 added Cl-36 detector efficiency calculation

19.5.8 Section 13.0 updated to include actual corrective actions and native concentration carrier requirements
19.5.9 Section 13.0 updated to include corrective actions
19.5.10 Section 17.0 added reference to ST-QA-0024

19.6 Rev. 15: (1/16/2015)
19.6.1 Added Section 7.10
19.6.2 Updated Section 9.6.1
19.6.3 Updated Section 10
19.6.4 Updated Section 11
19.6.5 Added ANSI N42.25-1997 reference to section 17

Title: ANALYSIS OF CYANIDE IN WATER AND SOIL [SW-846 9010C, 9012B; EPA 335.4]

Approvals (Signature/Date):

1/19/15 Kristen Ely — Date Inorganics Manager	1/16/15 Michael Ridenhower — Date Health & Safety Manager / Coordinator
1-16-15 Marti Ward — Date Quality Assurance Manager	Elaine Wild 2015.01.20 06:03:19 -06'00' Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-WC-0002 Rev. 16

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the determination of cyanide in drinking and surface waters, domestic and industrial wastes, soils/sediments and other solids.

1.2 This SOP is based on EPA method 335.4 and SW-846, 9010C and 9012B.

1.3 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 During the distillation, cyanide is converted to hydrogen cyanide vapor which is trapped in a scrubber containing sodium hydroxide solution.

2.2 The cyanide is then converted to cyanogen chloride by reactions with chloramine-T which subsequently reacts with pyridine and barbituric acid to give a red colored complex.

3.0 DEFINITIONS

3.1 See the St. Louis TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 CN, Cyanide - Defined as cyanide ion and complex cyanides converted to hydrocyanic acid by reaction in a reflux system of a mineral acid in the presence of magnesium ions.

3.3 CN, amenable- Defined as cyanides that are chlorinated which decompose at a pH less than 11.

4.0 INTERFERENCES

4.1 Most interference is removed by distillation prior to sample analysis.

4.2 The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser. When washing glassware, do not use soap. Rinse with hot water and reagent H_2O.

4.3 Thiocyanates may be an interference when present at high levels (> 10 mg/L).

4.4 Sulfides adversely affect the colorimetric procedures. Check for the presence of sulfides in the sample with lead acetate test paper. The paper will turn dark if sulfides are present. Notify PM before proceeding.

4.4.1 If sulfides are detected, treat the sample by adding 5 ml of bismuth nitrate solution through the air inlet tube after the air rate is set. Mix for 3 minutes prior to the addition of H_2SO_4. Note on the cyanide extraction sheet that this step was done. (For other cyanide methods, the sulfides are removed <u>after</u> distillation), **NOTE: Samples treated in this manner to remove sulfides must be accompanied by calibration standards which are also treated by the same procedure.**

4.4.2 For micro distillation refer to the operations manual to see how to make the trapping solution for sulfide positive samples. The calibration standards must also be distilled in the same manner.

4.5 Check for the presence of chlorine using KI-starch paper. If the paper turns dark, notify PM before proceeding. Samples are treated by adding ascorbic acid crystals a few at a time until strip produces no color. Then add an additional 0.06g ascorbic acid for each liter of sample volume.

4.6 Nitrate and/or nitrite may form oximes that will generate HCN, therefore all samples are treated for nitrate and/or nitrite. For Midi Distillation, add 5ml sulfamic acid solution after the air rate is set

through the air inlet tube. Mix for 3 minutes prior to the addition of H_2SO_4. For Micro Distillation, add 0.25 mL of the micro sulfamic acid solution just prior to the addition of the releasing agent. Note on the cyanide distillation prep sheet that this step was done.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 **SAFETY CONCERNS OR REQUIREMENTS**

5.1.1 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.2 PRIMARY MATERIALS USED

5.2.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

5.2.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m³ (Ceiling)	Inhalation symptoms may include coughing, sneezing, damage to the nasal and respiratory tract. High concentration can cause lung damage. Swallowing may cause serve burns of mouth, throat and stomach. Other symptoms may include vomiting and diarrhea. Serve scarring of tissue and death may result. Contact with skin can cause irritation or serve burns and scarring. Causing irritation of eyes with tearing, redness and swelling. Greater exposure can cause burns with possible blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 Mg/M3 (TWA) as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm (TWA)	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3- (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – time Weighted Average			
Ceiling – at no time should this limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Reflux distillation apparatus (Figure 1)

6.2 Lachat hot block

6.3 Boiling chips

6.4 Glass beads

6.5 Lead acetate paper/KI starch indicator paper

6.6 Beakers 50 or 100ml

6.7 Coarse glass fiber filter

6.8 High range pH paper

6.9 Suitable sample container, 50 ml centrifuge tubes

6.10 Technicon TRAACS 800, consisting of:

- 6.10.1 Random Access Sampler
- 6.10.2 Peristaltic Pump
- 6.10.3 Flowcell, 0.5 x 10 mm
- 6.10.4 Filter, 570 nm
- 6.10.5 Mutitest manifold
- 6.10.6 Computer for data collection, using New Analyzer Program (NAP), ver. 3.10.

6.11 Pipette, 10 ml, 1 ml and 100 µl

6.12 Volumetric containers

6.13 Graduated cylinder- 100 ml and 25 ml

6.14 Stir plate

6.15 Lachat collector tubes, user fill option or pre-filled.

6.16 Re-pipettor

6.17 Parafilm

6.18 Tube Press

6.19 Systea Easychem Plus Discrete Analyzer, consisting of:

- 6.19.1 Sample tray, using 2mL sample cups.
- 6.19.2 Reagent tray, capable of holding up to 18 reagents, temperature controlled.
- 6.19.3 Reagent cuvettes, temperature controlled cells where precise mixing of sample and reagents is performed.
- 6.19.4 Reagent and sample dosing station/dispensing syringe.
- 6.19.5 Colorimeter – 10mm path length flow cell with a 578nm optical filter.
- 6.19.6 Peristaltic pump.
- 6.19.7 PC for data collection, using Sysmedia Easychem software, ver.. 1.9.18

6.20 Printer

6.21 Analytical Balance

6.22 Scoopula

6.23 Heat resistant gloves

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards preparation, documentation and labeling must follow the requirements of ST-QA-0002.

7.2 NaOH 50% w/w, purchased commercially

7.3 NaOH, 1.0N: Dissolve 100 ml 10N of NaOH in distilled water and dilute to 1 liter or purchase commercially.

7.4 NaOH, 0.25N: Dissolve 25ml 10N of NaOH in distilled water and dilute to 1 liter or purchase commercially

7.5 Acetic Acid, glacial

7.6 Potassium Cyanide, reagent grade

7.7 Potassium Hydroxide, reagent grade

7.8 Sulfuric acid, concentrated

7.9 Stock QC Cyanide Standard: purchase commercially or prepare a 1000 mg/L CN by dissolving 2.51 g of potassium cyanide and 2.0 g of potassium hydroxide in one liter of DI water. Store under refrigeration. Stability is six (6) months or depends upon the manufacturer's recommendations.

7.10 Intermediate QC Cyanide Standard: for LCS/Low, High, and MS (10 mg/l): Dilute 1.0 ml of stock cyanide to 100 ml with 0.25 N NaOH. This standard should be preserved with 1 ml of 1.25 NaOH if the standard is prepared in advance. Stability is six (6) months or until parent stock expires.

7.11 Stock Cal Cyanide Standard: purchase commercially or prepare a 1000 mg/L CN by dissolving 2.51 g of potassium cyanide and 2.0 g of potassium hydroxide in one liter of DI water. Store under refrigeration. Stability is six (6) months or depends upon the manufacturer's recommendations. The standards must be from a different source than the QC stock.

7.12 Intermediate Cal Cyanide Standard (10 mg/l CN): To a 100 ml volumetric flask containing approximately 90 ml of 0.25N NaOH, add 1.0 ml of stock standard Bring to volume with 0.25 N NaOH and mix until homogeneous. Stability is six (6) months or until parent stock expires

7.12.1 Cyanide Calibration Curve: Using the intermediate cyanide standard and 0.25N NaOH for dilution, prepare the following working standards. The lowest standard must be at the RL or lower than the RL. Stability for each Calibration Curve point is 1 week.

Concentration of Working Standard (µg/L)	Aliquot of Intermediate (10 ppm) Standard (ml)	Dilution Volume (ml)
0	0	100
5	0.05	100
20	0.2	100
100	1.0	100
250	2.5	100
300	3.0	100
400	4.0	100
500	5.0	100

NOTE: If the samples were treated for sulfides before distillation, for method 9010, the calibration standards must be treated by the same procedure.

NOTE: The above intermediate cyanide standard is only used for calibration standards and CCV preparation.

7.13 ICV (200ug/L): Pipette 2ml of QC intermediate and bring to 100ml with 0.25 N NaOH, stability 1 week.

7.14 QC for Midi Distillation

7.14.1 High check standard (HLCS) (400 µg/L or 20mg/kg): Pipette 2 ml of the intermediate solution into a distillation setup containing 50 ml DI water (1g of glass beads to be included if soil prep). This will be used as a Distilled Check Standard (high) for Method 9010C, 9012B, and Method 335.4.

7.14.2 LCS/Low check standard (200ug/L or 10mg/kg): Pipette 1ml of the QC intermediate solution into a distillation setup containing 50ml of DI water (1g of glass beads to be included if soil prep). This LCS/Low will be used as a distilled check standard (low) for Method 9010C, 9012B and Method 335.4.

7.14.3 MS for Waters (200 µg/L): Pipette 1.0 ml of the QC intermediate solution into a distillation setup containing 50 ml of the designated sample

7.14.4 MS for soils (10 ug/g): Pipette 1.0 ml of the QC intermediate solution into a distillation setup containing approximately 1 gram of the designated sample that has been diluted to 50ml with DI water.

7.15 QC for Micro Distillation

7.15.1 High check standard (HLCS) (400ug/L or 4.8mg/kg): Pipette 6ml of the 400ug/L curve solution into a sample cup (0.5g of glass beads to be included if soil prep). This will be used as a distilled check standard (high) for Method 9010C, 9012B and Method 335.4.

7.15.2 LCS/Low check standards (200ug/L or 2.4mg/kg): Pipette 6ml of the 200ug/L ICV solution into a sample cup (0.5g of glass beads to be included if soil prep). This will be used as a distilled check standard (low) for Method 9010C, 9012B and Method 335.4.

7.15.3 MS for waters (200ug/L): Pipette 0.12mL of the QC intermediate solution into the sample cup containing 6mL of sample.

7.15.4 MS soils (2.4mg/kg): Pipette 0.12mL of the QC intermediate solution into 0.5g of sample and 5ml of DI water.

7.16 Magnesium Chloride ($MgCl_2 \times 6H_2O$), Reagent Grade.

7.17 Magnesium Chloride solution: Weigh 500 g of $MgCl_2 \times 6H_2O$ into a 1 liter flask, dissolve, and dilute to mark with deionized water.

7.18 Sulfamic acid, Reagent Grade

7.19 Sulfamic acid solution: Midi - Dissolve 40 g of sulfamic acid in DI water. Dilute to 1 liter
Micro – Dissolve 9.6g of sulfamic acid in DI water. Dilute to 100mls.

7.20 Deionized water, ASTM Type II.

7.21 Bismuth nitrate, reagent grade.

7.22 Bismuth nitrate solution: Dissolve 30.0 g of bismuth nitrate $Bi(NO_3)_3$ in 100 ml of DI water. Add an additional 500 ml of DI water. While stirring, add 250 ml of glacial acetic acid. Stir until dissolved. Dilute to 1 liter with deionized water.

7.23 Calcium Hypochlorite

7.23.1 Calcium Hypochlorite solution: Dissolve 5 g into 100 mL of DI water.

7.24 Ascorbic acid crystals.

7.25 Acetic acid, diluted 10X from glacial; carefully add glacial acetic acid to laboratory water in the ratio of one part acid to nine parts water.

7.26 Acetate buffer: Dissolve 410 g. sodium acetate trihydrate ($NaC_2H_3O_2 \bullet 3H_2O$) in 500 ml. water. Add glacial acetic acid to yield a solution with pH 4.5 (approximately 500 ml), giving a final volume of approximately 1 liter.

7.27 Zinc acetate solution, 100 g/L: Dissolve 120 g. Zinc acetate monohydrate [$Zn(C_2H_3O_2)_2 \bullet H_2O$] in 500 ml. reagent water. Smaller volumes of this reagent may be prepared using the same ratios of solid to water, or the solution may be purchased as a certified reagent. Prepare monthly.

7.28 Methyl red indicator: purchased commercially.

7.29 Barbituric acid, purified

7.30 Pyridine, reagent grade

7.31 Hydrochloric acid, concentrated

7.32 Deionized (DI) water, obtained from the Milli-Q unit.

7.33 NaOH, 10 N Solution, 50 % w/w, purchased commercially

7.34 Sodium Phosphate Monobasic ($NaH_2PO_4 \bullet H_2O$)

7.35 Brij-35, 30% solution

7.36 Cadmium carbonate, powdered.

7.37 Micro Distillation Releasing Agent

7.37.1 In a hood, place 110.8g of DI water into a 500ml beaker. Add and completely dissolve 32.2g of magnesium chloride ($MgCl_2$ x 6 H_2O). Add 139g (approximately 80ml) concentrated sulfuric acid SLOWLY (in roughly 20ml increments) while stirring. Let cool and add to the repipettor.

7.38 TRAACS Reagents

7.38.1 Pyridine-barbituric acid reagent

7.38.1.1 Place 7.5 g of barbituric acid into a 500 ml volumetric flask. Wash the walls of the flask with approximately 50 mls of DI water swirling flask to wet all of the barbituric acid. Next add 37.5 mls of pyridine using a 100 ml graduated cylinder. Add a magnetic stirring bar and begin mixing. While mixing, add 7.5 ml of concentrated hydrochloric acid with a graduated pipet and continue mixing until contents of the flask have dissolved. Remove the magnetic stirring bar, rinse the stirring bar with DI water (add rinsates to flask), bring to volume with DI water and mix until homogenous. Storage: Refrigerate at 4°C (±2°C). Stability: 1 month. May be purchased commercially.

7.38.2 NaOH, 1.0 N: Dissolve 32 ml of 50% w/w NaOH in DI water and dilute to 1 liter or purchase commercially.

7.38.3 Phosphate Buffer pH 5.2 ± 0.2 : To about 400 ml of DI H_2O add 69 g $NaH_2PO_4 \bullet H_2O$ (7.1.12). Stir until salts have dissolved. Dilute to 500 ml with DI H_2O. Add 1 ml Brij solution (7.1.13) and mix. Storage: Refrigerate at 4°C (± 2°C). Stability: 1 month.

7.38.4 Chloramine-T: Dissolve 2.0 g chloramine-T in about 400 ml of deionized water by swirling in a 500 ml volumetric flask. Bring to volume with deionized water and mix until homogeneous. Prepare fresh daily.

7.38.5 Brij Reagent – Pipette 1ml Brij 35 and dilute to 1L with DI water, stability: 1 month.

7.39 Discrete Reagent Preparation

7.39.1 Phosphate Buffer – add 13.8g sodium dihydrogen phosphate to a 130mL digestion tube and dilute to 100mL with DI. Stable for one month.

7.39.2 Chloramine – T Reagent – add 0.4g chloramine–t to 130mL digestion tube and dilute to 100mL with DI. Stable for one day.

7.39.3 Pyridine Barbituric Acid Reagent – in a fume hood, add 1.5g barbituric acid to a 130mL digestion tube, add about 50mL DI and begin to mix. Add 7.5mL pyridine and mix some more. Add 1.5mL concentrated HCI and dilute to 100mL. Stable for one month.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Water samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample (pH > 12) at the time of collection.

8.3 Oxidizing agents (chlorine) – Test a drop of sample with KI-starch paper. A blue color indicates treatment is needed. Add ascorbic acid (a few crystals at a time) until a drop of sample produces no color on the indicator paper. Add 0.6g of additional ascorbic acid for each liter of sample. This must be done by the sampler immediately after collection. Water samples must be preserved at the time of collection with 2ml of 10 N NaOH per liter (pH>12).

8.4 Method 335.4 requires the sample be checked for sulfide in the field and appropriate preservation be performed by the samplers. TestAmerica St. Louis does not collect samples. The laboratory will check for sulfide in the lab (Section 11). The project manager will notify the client if sulfide is found in the sample and request re-sampling.

8.5 Samples should be analyzed as soon as possible after collection. The maximum holding time for soil and water is 14 days from collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at 4°C ± 2°C.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), a High Level Laboratory Control Sample (HLCS), Matrix Spike (MS) and Sample Duplicate (SD). In the event that there is insufficient sample to analyze a MS, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across

QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A Method Blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is organic free water.

9.2.4 For Soil analyses, the method blank is comprised of boiling chips or glass beads.

9.3 **Laboratory Control Sample**

9.3.1 A LCS/HLCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS and HLCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS and HLCS is comprised of DI water fortified with Cyanide.

9.3.4 For Soil analyses, the LCS and HLCS is comprised of glass beads fortified with Cyanide.

9.4 **Sample Duplicates**

9.4.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision. For this method there should be one sample duplicate run per every ten analytical samples.

9.4.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.4.3 Additional Sample Duplicates do not count towards the 10 samples in an analytical batch.

9.5 **Matrix Spike/Matrix Spike Duplicate (Matrix Spike/Sample Duplicates)**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. For this method there should be one MS run per every ten analytical samples.

9.5.2 MS/MSD samples do not count towards the 10 environmental samples in a sample batch.

9.5.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.3 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibrate the balance daily when used. The balance is calibrated in accordance with SOP ST-QA-0005.

10.2 The TRAACS 800 instrument and the Discrete Analyzer must be calibrated every day before it is used for sample analysis. Include the instrument standardization date and time in the raw data.

10.3 The instrument is calibrated by analyzing a calibration blank and the calibration standards described in ascending order of concentration.

10.4 A linear calibration curve is plotted and a correlation coefficient calculated by the TRAACS 800software by comparing the standard concentration to the instrument response.

10.5 Instructions for programming the TRAACS 800 are contained in Section 11.

10.6 The calibration curve must have a correlation coefficient of 0.995 or greater.

10.6.1 Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. In such situations, the analyst should determine whether the analytical range should be decreased by deleting the high or low standard. If the low standard is deleted, adjust the reporting limit. If the high standard is deleted, dilute samples appropriately. If the high standard shows saturation of the response, the calibration range must be adjusted. |

10.6.2 Evaluate whether the problem was related to a misinjection of one of the standards. This must be confirmed by reanalyzing the suspect standard. If the reanalyzed standard is in line with the others, the misinjection of the original standard is confirmed. At this point, the analyst has the choice of deleting the offending calibration point, or replacing it with the new run. The minimum number of calibration points must be five.

10.7 Analyze an ICV immediately after the last calibration standard.

10.7.1 If the measured value of the ICV is not within 10% of the true value, for 300 series methods, ± 15% for SW846 methods, the analysis must be stopped, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.8 Analyze an ICB immediately after the ICV.

10.8.1 If the ICB or CCB result exceeds the reporting limit, stop analysis, correct the problem, recalibrate, verify the calibration and reanalyze all analytical samples analyzed since the last compliant calibration blank.

10.9 Analyze a CCV at the beginning of the run, every 10 samples during the sequence and after the last analytical sample.

10.9.1 The CCV and ICV must be ± 10% of true value. The high and low distilled check standard must be ± 10% for 300 series methods and ± 15% for SW846 methods.

10.9.2 If the CCV value is not within ± 10% of the true value for 300 series methods, and ± 15% for SW846 methods, stop analysis, correct the problem, recalibrate, verify the calibration and reanalyze all analytical samples analyzed since the last compliant CCV.

10.10 Analyze a CCB immediately after each CCV.

11.0 PROCEDURE

11.1 Pretreatment of samples for cyanide amenable to chlorination (perform in a hood):

11.1.1 Two sample aliquots are required to determine cyanide amenable to chlorination. To one aliquot of 50 ml add Calcium hypochlorite solution drop-wise while agitating and maintaining the pH between 11 and 12 with NaOH solution.

11.1.2 Test to ensure residual chlorine is present using KI-starch paper. A blue color on the paper indicates sufficient chlorine level.

11.1.3 After maintaining the chlorine level for one hour excess chlorine is removed by small additions of ascorbic acid (0.1g) until KI-starch indicator paper fails to give blue result. A small addition of ascorbic acid (0.1g) is then added to ensure chlorine removal.

11.2 MIDI DISTILLATION

11.2.1 For soils, accurately weigh a representative 1.00 g portion of wet sample (glass beads if method blank or LCS) and transfer it to the distillation setup. Add 50 ml of distilled water.

11.2.2 For liquids, place 50 ml of sample (deionized water if method blank), or an aliquot diluted to 50 ml in the distillation setup.

11.2.3 Test all samples for the presence of sulfide and chlorine. (see section 4.0)

11.2.4 The pH of liquid samples is checked by the Sample Control Dept at sample log-in.

11.2.5 Add 50 ml of 0.25N sodium hydroxide to the absorbing tube.

11.2.6 In LIMS, record the following information: date and time distilled, sample weights and volumes, QC batch number for prep blank and LCS, comments describing any sample reactions during preparation, and test strips results.

11.2.7 Add about 5-6 boiling stones to the boiling flask. Connect the boiling flask, condenser, absorber, and trap as indicated in Figure 1.

11.2.7.1 **Note:** the gas trap or vacuum exhaust trap should contain 0.25 N Na OH and be connected to the vacuum line.

11.2.8 Start a stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately 1.5 liters per minute enters the boiling flask (about 2-3 bubbles per second) through the air inlet tube. The bubble rate may not remain constant while heat is being applied to the flask. It may be necessary to re-adjust the air rate occasionally. (**Note: it is important that the solution in the boiling flask does not back up into the air inlet tube).**

11.2.9 Spike through air inlet and rinse with DI water.

11.2.10 For analyses other than Weak Acid Dissociable (WAD) cyanides, add 2mL of magnesium chloride solution and then add 5mL of sulfamic acid solution into the air inlet tube and allow the airflow to mix the contents for 3 minutes. Then slowly add 2mL of concentrated sulfuric acid and rinse the tube with DI water.

11.2.10.1 For WAD cyanides, add 2 ml. of acetate buffer and 2 ml. of zinc acetate solution through the inlet tube. Add 2 to 3 drops of methyl red indicator. Rinse the tube with water and let air mix the flask contents by bubbling. If the solution is not pink, add acetic acid (1:9) drop-wise through the inlet tube until a pink color persists.

11.2.11 Begin flow of cooling water to the cold finger condensers and heat the solution to boiling. Reflux for 1hour. Using the distillation block the heat will automatically be stopped and the samples allowed to cool. After cooling the boiling flask, disconnect absorber, close off the vacuum source and stop the cooling water.

11.2.12 For all samples drain the solution from the absorber into a suitable container. Centrifuge tubes may be substituted for volumetric flasks to contain the final sample distillate.

11.2.13 Transfer all distilled samples to the analysis section of the lab.

11.3 MICRO DISTILLATION

11.3.1 Warm Lachat hot block to 120 degrees C.

11.3.2 Place as many collector tubes as needed for the batch in the rack with the "D" end down and the "M" end up.

11.3.3 Pipette approximately 1.51 mL of 1.0N NaOH in M end of User-Fill collector tubes. Center a 30mm membrane over the "M" end and seat a cap firmly on the end. If using pre-filled collector tubes, put in rack with "M" end up.

11.3.4 Put sample tubes into the sample tube rack.

11.3.5 Place 6.0 mL of sample (or standard) into each sample tube

11.3.5.1 For soil/sludge add between 0.5 and 1.0 g of soil or sludge to 5.0 mL of water.

11.3.6 Test all samples for the presence of sulfides and chlorine. (see section 4.6,04.7 and 4.8)

11.3.7 In LIMS, record the following information: date and time distilled, sample weights and volumes, QC batch number for prep blank and LCS, comments describing any sample reactions during preparation, and test strips results.

11.3.8 Treat all samples for nitrates by adding 0.25mL of the sulfamic acid solution.

11.3.9 Prime the re-pipettor

11.3.10 Add 0.75 mL of the releasing agent to sample tubes.

11.3.11 Immediately push the "D" end of a Cyanide-1 collector tube over each sample tube and use a "tubepress" until the stop ring on the sample tubes hits the "D" end of the collector tube.

11.3.11.1 Grip collector tube at breakaway point to keep tube from shifting during pressing procedure. Use smooth constant pressure to avoid damaging the tubes.

11.3.12 Wearing heat resistant gloves, push the sample tube and "D" end of each tube completely down in the preheated block.

11.3.13 Set timer for 30 minutes.

11.3.14 After 30 minutes, wearing heat resistant gloves, remove the first tube from the block and immediately twist off the sample tube. **This must be done within 4 seconds to avoid "suck back".**

11.3.15 Dispose of acid solution in appropriate waste container. Discard sample tube into trash container.

11.3.16 Repeat this procedure for the rest of the sample tubes.

11.3.17 Invert all collector tubes and place them in the collector tube rack with the "D" end up and allow to cool for ten minutes.

11.3.18 Hold each tube horizontally and rinse the walls with the distillate. Slowly roll the tube to make the drops clinging to the walls settle down into the bulk of the distillate.

11.3.19 With the "D" end up, flick the tube with a finger to cause stubborn drops to fall into the "M" end.

11.3.20 With the "D" end up, break the collection tube by pulling the "D" end hard toward yourself. Twist off and discard the "D" end.

11.3.21 Place the collector tube rack with the "M" end down.

11.3.22 Dilute up to the 6.0 mL mark with DI water in the remaining "M" end, resulting in the original sample volume in 0.25M NaOH.

11.3.22.1 Cover the tube with parafilm and mix by shaking with a gently whipping motion.

11.3.22.2 Place the collector tube rack with the "M" end down.

11.3.22.3 Samples are ready for analysis.

11.4 ANALYSIS: The Traacs 800 system is a computer controlled, continuous flow wet chemistry analytical system using a colorimeter to detect changes in color produced by the presence of specific chemical substances in the analyzed samples. The operator interfaces with the system via the keyboard of an external computer. The basic system consists of the following modules: Random Access Sampler, Analytical Console, Computer with CRT Display, and printer.

11.5 NAP (New Analyzer Program)

11.5.1 TRAAC's 800 analysis (NAP – New Analyzer Program)

11.5.1.1 Secure all reagent tubing by making sure both stops of the tubes are stretched between the notches in the peristaltic pump. Close the plate and secure with the clamps.

11.5.1.2 Place the ends of the tubing into the respective reagent bottles.
11.5.1.3 Double click on the NAP icon.
11.5.1.4 Select 'Control' in the menu, choose the correct channel and click the 'Normal' button under Status & Control to start up the instrument.
11.5.1.5 In the channel for the appropriate analysis, click on the arrow icon in the sample box and select the proper template (CNxxxxxx.spl)
11.5.1.6 Using the barcode scanner and the 8 digit barcode report, scan all samples into the sample table (inserting a CCV/CCB every 10 samples), add in the correct location for each sample and save as the analysis and the date (i.e CN041111)
11.5.1.7 Once a stable bubble pattern has been established, close out of NAP, open it up again, enter the save file name into both the Sample Table and Data columns, and click 'Standby' to begin the automatic base and gain
11.5.1.8 Click the mouse in the Sample Table box, then click 'View' to see the base and gain correction
11.5.1.9 Once that is complete, the screen will disappear and 'View' must be selected again to see the run data
11.5.1.10 If, during the run, a sample comes out above the highest calibration point, a dilution can be made and added to the sample table as long as everything has not already been sampled (everything will be grayed out if all injections have been made)
11.5.1.11 When the run is complete and the blue baseline correction line has been drawn, take all of the ends of the tubing out of the reagent bottles and put each one (with the exception of the rinse line tubing on the far right) into a container that has a solution of Brij water and DI. Continue to let the instrument flush until the bubble pattern has been disrupted throughout the lines
11.5.1.11.1 Review the data for appropriate peak shape and corresponding instrument response. Occasionally, the system fails to identify a peak or mislabels a peak. If a manual integration is performed, it should be documented on the raw data with the reason for integration along with initials and date.
11.5.1.11.2 In the view table, select 'Print Results' and print. Under 'Standards,' select print. Then select 'Print' in the main analyzer window.
11.5.1.11.3 To upload the file, click on 'Reporting," then click 'Output to ASCII File," Next, click the File button and change the drive to G:. In the list of Folders, select Wet Chem, then select the TR1 instrument. Next, double click on the method to be uploaded (ex: 353.1), and rename the file to reflect the analysis and date, followed by ".TRX" at the end of the file name. (ex: N370114.TRX." Then click "OK," then click "Output," and the file is uploaded to LIMS. ,

11.5.1.12 Turn the instrument off by selecting 'Control' in the menu, choosing the correct channel, and clicking 'Off.' You may have to close NAP again and restart first in order for it to turn off.
11.5.1.13 Unclamp the plate covering the reagent tubes, and pull the bottom stops out from the notches to reduce wear on the tubing.

11.6 Instrument Startup and Analysis – Systea Instrument
11.6.1 Power on the Easychem unit. Select the Easychem icon to open the data acquisition software. Login to the software. When the system has been properly initialized, a message box displaying "Instrument ready" will appear.
11.6.2 Fill the rinse water container with new reagent grade water. Tighten the peristaltic pump winding.
11.6.3 Empty the waste container.

11.6.4 Through the "User Service" tab, have the instrument perform 15 washes, followed by the filter's autozero function. New values for all filters must match within 5% of the old values. If they do not meet the criteria, the instrument requires maintenance.

11.6.5 Through the "Operations" tab, and "Worklist Editor," create a sample worklist with the appropriate method applied to each sample. Load the samples, calibrants, QC, LCS, MB, etc., into the autosampler tray.

11.6.6 Prepare the working reagents. Fill the R1 (Phosphate Buffer), R2 (Chloramine-T), and R3 (Pyridine Barbituric Acid) reagent containers and place in the correct cartridge position as specified in the instrument method (R1 is position R8, R2 is position R9, and R3 is position R10). Place the cartridge into the reagent tray on the analyzer.

11.6.7 Through the "Operations" tab select "Start Analysis". Check to ensure that the current worklist is loaded. Change both the analysis file (.slz file) and the analysis text file (.txt file) locations and name to the type and date of the analysis (i.e. CN 041111).

11.6.7.1 Verify that there are sufficient reaction cuvettes to complete the run. Replace if needed, and change the starting location.

11.6.7.2 Verify that reagent and cuvette temperatures are within control. (Instrument will monitor temp and alert analyst to excursions.) Start the analysis.

11.6.7.3 To export results, click "File," "Export Results," then "Continue." Save the file as the analysis name, followed by the date (ex: CN051414). At the top of the window, under "Save In" select "Easychem," followed by "TALS Export" and click save. Then exit out of the program and open the desktop folder "TALS Export," right click on the file that was just saved, and select "Send To," and select the appropriate method being uploaded. The data is now uploaded to LIMS.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the St. Louis ST-QAM.

12.2 The TRAACS 800 data system will prepare a standard curve by plotting peak heights of standards against concentration values. It will then compute the concentration of samples by comparing sample peak heights with standards.

12.3 Final Concentration (water)

$$\text{Final Concentration } (\mu g/L) = \frac{(RV \times D)}{V_O} \times V_D$$

where:
RV = Raw value (μg/L) from instrument
D = Dilution factor, if any
V_D = Distillate Volume (L)
V_O = Original volume (L)

12.4 Final Concentration (soil)

12.4.1 If the concentration for soil is on dry weight basis, determine the solid fraction of the sample. Refer to SOP STL-WC-0005, Percent Solids Determination.

$$\text{Final Conc } (\mu g/g) = \frac{(RV \times D)}{W \times SF} \times V_D$$

where:
W = Weight of sample (g)
SF = Solid Fraction (%Solids/100)
And remaining terms are as defined in 12.1

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.

13.2.1.1 Project specific requirements, if more stringent than the lab routine procedure (e.g. no target analytes present above ½ RL), will be noted in the comments section of LIMS.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the samples must be re-prepped/re-analyzed unless their concentration is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in the LIMS.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM, (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.3.2.3 RPD excursion for LCS/LCSD – If spike recoveries are acceptable, data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes.

13.4 Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/SD)

13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in LIMS.

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.4.2.1.1 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.4.2.1.2 If the excursion is due to a physically evident matrix interference, the data is reported with an NCM.

13.4.2.1.3 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with and NCM.

13.4.2.1.4 When the MS/MSD recoveries and the %RPD are outside criteria, the batch is re-prepped and re-analyzed for the affected analytes.

13.4.2.2 % RPD excursion for MS/MSD - sample/sample duplicate – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM describing the physical evidence; otherwise the sample should be re-prepped and re-analyzed

13.5 Sample result evaluation

13.5.1 Dilutions

13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.5.1.1.1 An NCM is written to document the reason for the dilution.

13.5.1.1.2 If the dilution causes the Spike(s) to be diluted out an NCM is written to document the excursion.

13.6 Insufficient Sample

13.1.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Wastewater contaminated with Pyridine shall be collected in a 5-gallon poly waste accumulation container, labeled as "Waste Pyridine". Please refer to SOP ST-HS-0001 and ST-HS-0004 for proper waste handling.

17.0 REFERENCES

17.1 Standard Methods for the Examination of Water and Wastewater, 17th Edition, Method 4500-CN.

17.2 EPA Manual 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983. Method, 335.4.

17.3 EPA SW-846, Update IV, Method 9010C and Method 9012B November 2004

17.4 Industrial Method No. 80286T from Technicon TRAACS 800 Method.

17.5 "Micro Distillation Operations and Applications", Lachat Instruemts Catalog 01304, HACH Co. 2004 – 2006

17.6 "Easy Chem Methodology", Systea Scientific, LLC, February 2009

17.7 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.8 St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.9 Associated SOPs (current revisions):
- 17.9.1 ST-PM-0002, Sample Receipt and Chain of Custody
- 17.9.2 ST-QA-0002, Standards Preparation.
- 17.9.3 ST-QA-0011, Data Review, Verification and Reporting.
- 17.9.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process.
- 17.9.5 ST-WC-0005, Percent Solids Determination.
- 17.9.6 ST-IP-0004, Glassware Preparation for Inorganic and Trace Metals Analysis
- 17.9.7 ST-QA-0005, Calibration and Verification Procedure for Laboratory Support Equipment
- 17.9.8 ST-HS-0001, Waste Minimization Plan
- 17.9.9 ST-HS-0004, Hazardous Waste Management Plan

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCED METHODS

18.1 The SW846 9000 series methods for Cyanide analysis require that the Method of Standard Addition (MSA) be used for samples with matrix interferences. TestAmerica St. Louis does not perform an MSA. For samples demonstrating matrix interference, the sample is diluted to try to eliminate the interference and an NCM is included in the case narrative.

18.2 Amounts of standards and reagents used may vary from the method guidance. QC check samples, MDL studies and PT sample results demonstrate that the changes have not adversely impacted the results of the analysis.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated section 4.0 regarding PM notification.

19.2 Rev. 14:
- 19.2.1 Updated title of Sop from "Cyanide Analysis by the Technicon Traacs 800 Auto Analyzer" to "Analysis of Cyanide in Water and Soil".
- 19.2.2 Updated the Method procedural numbers to 9010C and 9012B throughout SOP.
- 19.2.3 Added instructions for Micro Distillation treatment of sulfide interferences to Section 4.
- 19.2.4 Removed Phosphoric Acid and Silver Nitrate from the safety table in section 5.0.
- 19.2.5 Added Systea Easychem Plus Discrete Analyzer and its supplies to section 6.0.
- 19.2.6 Added a stock cal standard along with instructions to section 7.0.
- 19.2.7 Inserted a Traacs Reagent section, 7.44.
- 19.2.8 Inserted a Discrete Reagent Preparation section, 7.45.
- 19.2.9 Corrected SOP reference for balance calibration procedure from ST-WC-0005 to ST-QA-0005 in section 10.0.

19.2.10 Removed the requirement for the undistilled ICVS from Section 10.
19.2.11 Noted that the pH check is done at time of receipt by the Sample Control Dept in section 11
19.2.12 Updated Section 11.5 with new NAP instructions.
19.2.13 Created Section 11.6 for start-up and analysis using the Systea Instrument
19.2.14 Updated Reference Section (17) to include method revisions and new instrument documentation.
19.2.15 Updated the Cyanide Distillation Log worksheet (Attachment #2).

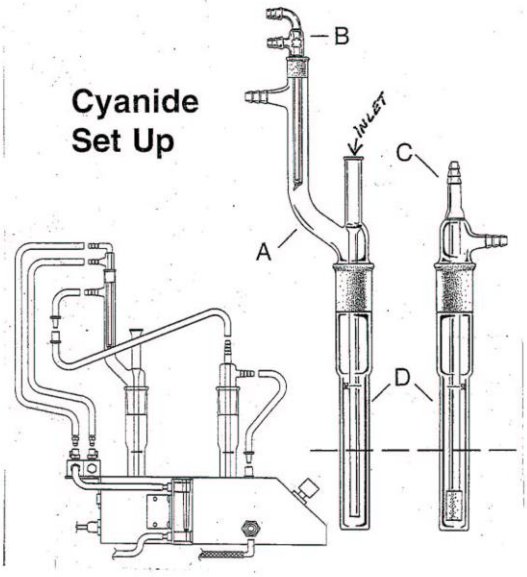
19.3 Rev. 15:
19.3.1 Updated section 7.0 regarding the amounts of NaOH used.
19.3.2 Updated the amount of NaOH used during pipetting for micro-distillation in section 11.3.
19.3.3 Updated information for pre-filled collection tubes in section 11.3.23.

19.4 Rev. 16: (6/18/2013)
19.4.1 Removed references to “Clouseau” and “QuantIMS” replacing with “LIMS”.
19.4.2 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.
19.4.3 Updated supplies in section 6.0.
19.4.4 Removed the use of Sodium Hypochlorite throughout the entire SOP and replaced it with Calcium Hypochlorite.
19.4.5 Updated section 13.0.
19.4.6 Updated section 15.0.

19.5 Rev 17: (1/16/2015)
19.5.1 Updated Inorganics Manager on cover page
19.5.2 Added Glass Beads to section 6
19.5.3 Reorganized Cal and QC stocks and their intermediates in section 7.
19.5.4 Clarified throughout the SOP that the High Check Standard or HCS is now called the HLCS in LIMS.
19.5.5 Removed redundancies throughout the SOP
19.5.6 Removed references to the distillation log from section 11 and also removed the excel spreadsheet for this log from the last page of the SOP.
19.5.7 Updated the correct upload procedures for the TRAACS and Systea in sections 11.5 and 11.6.
19.5.8 Added three Associated SOPs to section 17.9

Attachment 1

Figure 1 -



Kible - Konte Glassware

Cyanide Distillation Apparatus, Complete, MIDI-VAP™ 2000

Fisher Article No.	Description	Case Qty.	Case Price
K479200-2000	**MIDI-VAP™ 2000, Cyanide Complete Includes the following:**	1	6,789.51K
K479160-0000	Midi-Cyanide Glassware (479200-2000 supplied with 10 sets)	1	246.14
K720140-2000	MIDI-VAP™ 2000 Manifold Heater, 115-V	1	4,841.53
Component Parts			
K479161-0000	(A) Head, ₮ 29/42 Lower, ₮ 14/20 Upper	1	96.90
K282000-0000	(B) Cold Finger Condenser, ₮ 14/20, #1 Hose Connection	1	47.11
K479162-0023	(C) Dispersion Tube, Coarse Porosity, ₮ 29/42, #1 Hose Connection	1	70.45
K479155-0050	(D) Purge Tube, ₮ 29/42 (2 Supplied)	1	42.10

Title: ANALYSIS OF SULFIDE
[SW-846 9034; EPA 376.1; Standard Method 4500 S^2 C and F]

Approvals (Signature/Date):

Kristen Ely 10/21/14 Kristen Ely Date Inorganics Department Manager	Michael Ridenhower 10/20/14 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 10-21-14 Marti Ward Date Quality Assurance Manager	Elaine Wild 10/29/14 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-WC-0012 Rev. 12

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.

1.2 This method is also applicable to the measurement of water extractable sulfides in soil/sediments and other solids.

1.3 This method is suitable for measurement of sulfides in concentration above 1 mg/L for waters.

1.4 This method is suitable for measurement of sulfides in concentration above 10 µg/g for soils. The factors which may make this limit higher are the percent moisture for sample and the exact aliquot used in analysis.

1.5 This SOP is applicable to EPA Method 376.1, SW-846 Method 9034 and Standard Methods 4500 S^{2-} C and F.

1.6 The distillation procedure, SW-846 Method 9030B is described in SOP: ST-IP-0019.

1.7 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the appendix of this SOP.

2.0 SUMMARY OF METHOD

2.1 The sulfide is oxidized to sulfur by adding a known excess amount of iodine. The excess iodine is determined by titration with a standard solution of phenyl arsine oxide (PAO) or sodium thiosulfate until the blue iodine starch complex disappears. As the use of standard sulfide solutions is not possible because of oxidative degradation, quantitation is based on the PAO or sodium thiosulfate.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Reduced substances that react with iodine, such as sulfite, thiosulfate and hydrosulfite, will decompose in acid and yield erratic results.

4.1.1 These interferences are eliminated by precipating Zinc sulfide, removing the supernatant and replacing it with DI water.

4.2 Volatile iodine consuming substances will give high results.

4.3 Sample distillation interferences are found in SOP: ST-IP-0019.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and

reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. Inhalation of HS gas may be fatal.

5.2 PRIMARY MATERIALS USED
The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Sign and Symptom of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Contact can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Sulfide	Corrosive	10 ppm (TWA) 15 ppm (STEL)	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average;			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Filtering Flasks.

6.2 Graduated cylinder

6.3 Erlenmeyer Flasks - 250 ml, or equivalent

6.4 Digestion tube, 130mL

6.5 Disposable cups, 100mL

6.6 Pipettes – adjustable eppendorfs, or equivalent.

6.7 Disposable Pipettes.

6.8 Glass Fiber Filter, 0.45 pm.

6.9 Volumetric Flasks - 100 ml, 500 ml and 1000 ml.

6.10 Balance, analytical.

6.11 pH paper, narrow range

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revisions.

7.2 DI water, obtained from the Millipore unit.

7.3 Potassium Iodide.

7.4 Standard iodine solution, 0.0250 N, commercially available

7.4.1 Dissolve 20 to 25 g Potassium Iodide in DI water in a 1 liter volumetric flask and add 3.2 g iodine (I_2). Allow to dissolve. Dilute to the mark with DI water and standardize against 0.0250 N PAO using a starch indicator.

7.5 Sodium Thiosulfate, 0.0250 N or 0.010 N, commercially available

7.5.1 Dissolve 6.205 g $Na_2S_2O_3$,-5H_2O in approximately 500 ml DI water in a 1 liter volumetric flask. Add 1.5 ml 6 N NaOH and dilute to the mark with DI water. Use a starch solution as indicator during titration. Also commercially available.

7.6 Starch indicator, commercially available.

7.7 Zinc acetate solution

7.7.1 Dissolve 220g Zinc acetate in 870ml water.

7.8 Sodium Hydroxide, NaOH, 6N, commercially available

7.9 Hydrochloric acid, HCl, 6N, commercially available.

7.10 Sulfide Solution: 1,000 mg/L, commercially available. Dissolve approximately 7.54 g of Na_2S-9H_2O in 1 liter of deionized water to yield a concentration of approximately 1000 mg/L of S (sulfur conversion factor = 0.133508).

7.10.1 Standards must be prepared at pH > 9 and < 11.

7.10.2 Shelf life: Standard must be made fresh **DAILY**.

7.10.2.1 LCS and Matrix Spike: For waters, spike the LCS and sample into the 500 ml flask with 2 mls of the sulfide solution. Use 200 mls water for the LCS. The true value

for the LCS and matrix spike (waters) is 10 mg/L. For the LCS and matrix spike (soils), the true value is 200 mg/kg.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 See Sample Preparation SOP, ST-IP-0019 for preservation requirements.

8.3 If not immediately analyzed after distillation, store distillate in refrigerator at 4°C ± 2°C.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Method Blank, (MB) a Laboratory Control Sample (LCS), Matrix Spike (MS) and a Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.4 **Matrix Spike (MS)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 **Sample Duplicate (SD)**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 The LCS serves as calibration check.
- 10.1.1 Frequency: An LCS or CCV is analyzed at the beginning and end of the analytical sequence.

10.2 Standardize the 0.025N I_2 weekly, if not purchase commercially or required by client.
- 10.1.1 Add 10 mls 0.025N Iodine to 200 mls water.
- 10.1.2 Titrate the solution using 0.025N Sodium thiosulfate. Repeat two more times and average mls titrant.
  - 10.1.2.1 Calculate the normality as follows:

    $$\text{Normality } (I_2) = \frac{\text{ml of titrant x normality of titrant}}{\text{sample size in ml}}$$
  - 10.1.2.2 Standardization of the iodine solution will be performed with each analytical batch or at a maximum frequency of once per week.

10.2 No standardization is required for the iodine solution if purchased commercially unless specifically unless specifically required by the client.
- 10.2.1 If a commercial standardized solution is purchased, this standardization step is not required.
- 10.2.2 Standardize against Potassium bi-iodiate after at least two weeks from preparation.
  - 10.2.2.1 Dissolve 3.249g of Potassium bi-iodate in DI water. Dilute with DI water to a 1000ml.
    - 10.2.2.1.1 This yields a 0.1N $KH(IO_3)_2$ solution.
  - 10.2.2.2 In a 250ml flask, add 1ml Sulfuric acid and 10ml $KH(IO_3)_2$, and dilute with DI water to 100ml.
  - 10.2.2.3 Place flask on stirring plate and stir.
  - 10.2.2.4 Add 1g Potassium iodide
  - 10.2.2.5 Titrate with 0.1 Sodium thiosulfate until the yellow color is almost discharged.
  - 10.2.2.6 Add 1ml of starch indicator and continue titrating until the blue color disappears.
  - 10.2.2.7 Normality (Sodium thiosulfate) = $\frac{\mathbf{1}}{\text{ml of tritrant}}$

11.0 PROCEDURE

11.1 376.1 Solid and Liquid
- 11.1.1 Unprecipitated Sample
  - 11.1.1.1 Place 10ml of standard iodine solution into a 500ml flask. The amount should be estimated to be in excess of the amount of sulfide expected.
  - 11.1.1.2 Add distilled water, if necessary, to bring the volume to approximately 20mls.
  - 11.1.1.3 Add 2 ml of 6N HCL
  - 11.1.1.4 For liquid samples, pipette 200 ml of sample into the flask, keeping the tip of the pipette below the surface of the sample.
    For solid samples weigh 20g of sample into a 130mL digestion tube add 100ml of DI and place on shaker for 10min. let particulates settle for approximately 2hrs. pipette 50mls of sample into a 500ml flask, keeping the tip of the pipette below the surface of the sample.
  - 11.1.1.5 If the iodine color disappears, add a few drops of 6N HCL. Addition of the sample may cause the pH to rise. If iodine color is still not present, add more iodine until the color remains recording the total volume of iodine used.
  - 11.1.1.6 Titrate with 0.0250 N sodium thiosulfate using a starch indicator until the blue color disappears. Record the number of milliliters used.

11.2 Precipitated samples liquid samples

11.2.1 Add the reagent to the sample in the original bottle. Perform steps 1, 3, 5 and 6 from above.

11.3 Pretreatment of Water samples (SM 4500-S_2 C and F)

11.3.1 For each sample and QC perform the following:
11.3.2 Place 1.0ml Zinc Acetate solution and 0.50 ml 6N NaOH in a 500ml bottle
11.3.3 Fill to a 100ml with sample and stopper bottle. Be sure there are no air bubbles.
11.3.3.1 For method blanks, fill to 100ml with DI water
11.3.3.2 For LCS, fill to 100ml with prepared LCS solution.
11.3.4 Mix sample vigorously.
11.3.5 Filter sample.
11.3.6 Collect precipitate on a glass fiber filter.
11.3.6.1 Store filter in original 500ml bottle.
11.3.7 Proceed immediately to the titration step.

11.4 Analysis of Sulfide Distillate (SW 9030B and SW 9034), and Pretreated Samples (SM 4500-S_2 C and F)

11.4.1 The sulfide distillation procedure is found in ST-IP-0019.
11.4.2 Clean burette and flask in accordance with cleaning procedure SOP, ST-IP-0004.
11.4.3 <u>Acid Soluble Sulfide sample analysis (SW 9034)</u>:
11.4.3.1 For each sample and QC perform the following:
11.4.3.2 Add 65ml iodine solution to titration flask.
11.4.3.3 Add 2ml 6N HCl to titration flask.
11.4.3.4 Pipette the Zinc Acetate scrubbing solutions obtained in Method 9030 to the flask, keeping the end of the pipette below the surface of the iodine solution. The entire scrubber solution is used.
11.4.3.4.1 If in transferring the solution the amber color of the iodine disappears or fades add more acid, sometimes the sample will raise the pH. Extra iodine may not be need but if iodine is needed, record the volume, 0.025N iodine.
11.4.3.5 Record the total volume of standardized 0.025N iodine solution used.
11.4.3.6 Rinse scrubber tube with a known amount of standardized 0.025N iodine solution, 1mL of 6N HCI, and water.
11.4.3.6.1 This rinses the remaining white precipitate (zinc sulfide) from the gas scrubbing bottles into the flask. There should be no visible traces of precipitate after rinsing.
11.4.3.7 Final volume in titration flask should be at least 100ml.
11.4.3.8 If volume is not at least 100ml, add DI water to bring up to 100ml.
11.4.3.9 Proceed to titration step 11.1.1.6.

11.4.4 <u>Acid Insoluble Sulfide sample analysis (SW 9034)</u>:
11.4.4.1 For each sample and QC perform the following:
11.4.4.2 Add 65ml iodine solution to titration flask.
11.4.4.3 Add 10ml 6N HCl to titration flask.
11.4.4.4 Pipette the gas sulfide Zinc Acetate scrubbing solutions obtained in Method 9030 to the flask, keeping the end of the pipette below the surface of the iodine solution. The entire scrubber solution is used.
11.4.4.4.1 If in transferring the solution the amber color of the iodine disappears or fades, add more 0.025N iodine.
11.4.4.5 Record the total volume of standardized 0.025N iodine solution used.
11.4.4.6 Rinse scrubber tube with a known amount of standardized 0.025N iodine solution, 1mL of 6N HCI, and water.
11.4.4.6.1 This rinses the remaining white precipitate (zinc sulfide) from the gas scrubbing bottles into the flask. There should be no visible traces of precipitate after rinsing.
11.4.4.7 Final volume in titration flask should be at least 100ml.
11.4.4.8 If volume is not at least 100ml, add DI water to bring up to 100ml.

11.4.4.9 Proceed to titration step 11.1.1.6.

11.4.5 Sulfide analysis (SM 4500-S_2 F):

11.4.5.1 For each sample and QC perform the following

11.4.5.2 Add 20ml iodine solution to titration flask.

11.4.5.3 Add 2ml 6N HCl to titration flask.

11.4.5.4 Pipette 200ml pretreated sample, keeping the end of the pipette below the surface of the iodine solution.

11.4.5.4.1 If in transferring the solution the amber color of the iodine disappears or fades, add more 0.025N iodine.

11.4.5.5 Record the total volume of standardized 0.025N iodine solution used.

11.4.5.6 Proceed to titration step 11.1.1.6.

11.4.6 Analysis of Sulfide Distillate obtained by micro distillation

11.4.6.1 Add 10mL of deionized water and 2mL of Iodine solution to the analysis cup.

11.4.6.2 Allow tubes to cool for at least 10 minutes after completion of the distillation.

11.4.6.3 Hold the tubes horizontally and rinse walls with distillate. Slowly roll the tube to make drops clinging to the walls settle down into the bulk of the distillate.

11.4.6.4 With the "D" end up, break the collection tube by pulling the "D" end hard towards yourself.

11.4.6.5 Rinse the "D" end into the analysis cup with deionized water making sure to rinse all sides of the tube. Rinse an additional two times with DI water.

11.4.6.6 Use a transfer pipette to inject the distillate in the "M" end of the collection tube below the surface of the liquid in the analysis cup. Repeat 11.4.6.5 for rinsing any residual distillate from the "M" end of the tube.

11.4.6.6.1 If in the transferring the solution the amber color of the iodine disappears or fades add more acid, sometimes the samples will raise the pH. Extra iodine may not be needed but if iodine is needed record any additional volume of iodine.

11.4.6.7 **Note:** when rinsing the collection tubes use minimal amount of DI water necessary. Over dilution record any additional volume of iodine.

11.4.6.8 Analyze the sample using a 10mL burette.

11.5 Titration

11.5.1 Titrate the solution with standardized 0.025N or 0.010N sodium thiosulfate solution until the amber color fades to yellow.

11.5.2 Add starch indicator for the solution to turn dark blue, titrate until the blue disappears.

11.5.3 Record the volume of titrate used.

11.5.4 For SM 4500-S_2 F (only):

11.5.4.1 Take 500ml bottle containing precipitate and filter.

11.5.4.2 Add 20ml iodine solution to titration flask.

11.5.4.2.1 If in transferring the solution the amber color of the iodine disappears or fades, add more 0.025N iodine.

11.5.4.3 Record the total volume of standardized 0.025N iodine solution used.

11.5.4.4 Add 2ml 6N HCl to titration flask.

11.5.4.5 Titrate as described in 11.1.1.5 – 11.1.1.6.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis QAM.

12.2 For water samples use the formula:

$$\text{Sulfide mg/L} = \frac{[(\text{ml x N iodine}) - (\text{mL x N } Na_2S_2O_3)] \text{ x } 16000}{\text{mL of sample}}$$

where N = normality of iodine or $Na_2S_2O_3$

12.3 For soil samples use the formula:

$$\text{Sulfide } \mu g/g = \frac{[(\text{mL x N iodine}) - \text{mL x N } Na_2S_2O_3)] \text{ x } 16000 \text{ x Ext vol original (mL)}}{(\text{Ext vol titrated, mL}) \text{ x } (\text{g of sample}) \text{ x } (\text{solid fraction})}$$

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Clouseau nonconformance memorandum (NCM) process. The NCM process is described in sop: ST-QA-0036. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlayte present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 <u>Method Blank Contamination</u> – See Clouseau NCM for corrective action (e.g. reprep/reanalysis, narration). Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in Clouseau.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 <u>LCS Spike Recovery excursion (high)</u> – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

13.3.2.2 <u>LCS Spike Recovery excursion (low)</u> – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

13.3.2.3 <u>RPD Duplicate excursion</u> – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 <u>MS/MSD Spike Rec. excursion</u> may not necessarily warrant corrective action other than narration. See Clouseau NCM to determine if re-preparation re-analysis is required.

13.5 Insufficient Sample

13.5.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis and narrative comment stating such is included in the report narrative. The insufficient sample description is included in the Clouseau NCM within the type defining the excursion.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.

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14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out

16.2.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 EPA Manual 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Method 376.1.

17.2 Standard Methods for the Examination of Water and Waste Water, 19th Edition 1995, Method 4500-S_2-C and Method 4500-S_2-F.

17.3 SW-846, Method 9030B Rev 2 1996 and 9034 Rev 0 1996, Sulfides, Third Edition

17.4 TestAmerica Quality Assurance Manual (QAM), current revision

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.4 Associated SOPs – current revisions
- 17.4.1 ST-IP-0004, Glassware Preparation for Inorganic and Trace Metals Analysis
- 17.4.2 ST-IP-0019, Sulfide Distillation
- 17.4.3 ST-QA-0002, Standard and Reagent Preparation
- 17.4.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.4.5 ST-QA-0016, IDL/MDL Determination
- 17.4.6 ST-QA-0036, Non-conformance Memorandum (NCM) Process
- 17.4.7 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 CLARIFICATIONS, MOIDIFICATIONS TO THE REFERENCE METHOD.

18.1 None.

19.0 CHANGES TO PREVIOUS

19.1 Added method 376.1 to the section 11.0.
19.2 Updated section 7.0 by changing the amount sulfide used in a LCS for waters.
19.3 Removed Low-levels and High-Levels LCS from section 10.0.
19.4 Rev, 10. Added digestion tubes and disposable cups to section 6.0.
19.5 Rev, 10. Added 1,000 mg/L of commercial grade sulfide solution to section 7.0.
19.6 Rev, 10. Increased the amount of sulfide solution standard from 250 to 500mls and 100 to 200 mls in section 7.10.2.
19.7 Rev, 10. Increased the flask size from 250mls to 500mls in section 11.1.1.4.
19.8 Revision 11:
- 19.8.1 Updated section 11.0 by adding instructions for Analysis of Sulfide Distillate obtained by micro distillation.

19.9 Revision 12:
- 19.9.1 Updated titration reference step in sections 11.4 and 11.5.

19.10 Revision 13:
- 19.10.1 Updated reagent Sodium Thiosulfate in section 7.5
- 19.10.2 Updated micro distillation analysis procedure in section 11.4.6.
- 19.10.3 Updated titration procedure in section 11.5.

Attachment 1

TestAmerica St. Louis Laboratory
SULFIDE

Analyst: _____
Analysis Date: _____
Reviewed By: _____
Date: _____

Method No.: _____
Batch No.: _____

A sample and its filter must be entered on two adjacent lines of the spread sheet. Sample first, filter on line right below.

Laboratory ID	Filter Y/N	True Value mg/L	Sample Vol., mL	Iodine (mL)	Titrant, mL $Na_2S_2O_3$ Initial	Final	Net	Sulfide mg/L	Samp + filt Sulfide mg/L	Percent Recovery	RPD

Iodine (I_2) Normality (N) = 0.025
$Na_2S_2O_3$ Normality (N) = 0.025

$$\text{Sulfide mg/L} = \frac{[(\text{mL } I_2 \times \text{N } I_2) - (\text{mL } Na_2S_2O_3 \times \text{N } Na_2S_2O_3)] \times 16{,}000}{\text{mL Sample}}$$

SOP	Rev	Date	Control Limits (Water)		Control Limits (Soil)	
STL-WC-0012	5	6/20/07	LCS = 75-112	MS = 68-128	LCS = 85 - 105	MS = 76 - 106

File: Slsvr01\public\inorganics\Wet Chemistry\spreadsheets\sulfide, modified 6/19/07

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Title: ALKALINITY IN WATER AND SOIL
[EPA 310.1; Standard Method 2320B]

Approvals (Signature/Date):

Kristen Ely — 5/11/15 — Date Inorganics Department Manager	Michael Ridenhower — 5/11/15 — Date Health & Safety Manager / Coordinator
Marti Ward — 5.11.15 — Date Quality Assurance Manager	Elaine Wild — 5/11/15 — Date Laboratory Director

This SOP was previously identified as SOP No. ST-WC-0019 Rev. 15

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the measurement of alkalinity in drinking, surface, saline waters, domestic and industrial wastes, and water extractable alkalinity of soil/sediments and other solids.

1.2 Total alkalinity and the approximate concentration of carbonate, hydroxide, and bicarbonate alkalinity can be determined.

1.3 Report alkalinity less than 20 mg $CaCO_3$/L only if it has been determined by the low alkalinity method.

1.4 This SOP is applicable to EPA Method 310.1 and SM 2320B.

1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 A sample is titrated to an electrometrically determined end point of pH 8.3 for phenolphthalein alkalinity, pH 4.5 for total alkalinity and pH 4.2 for low alkalinity.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Total Alkalinity (T): Sum of all the titratable bases.

3.3 Phenolphthalein Alkalinity (P): A quantity measured by titration to pH 8.3 during alkalinity determination.

3.4 Hydroxide Alkalinity (OH^-): Present if phenolphthalein alkalinity is less than half the total alkalinity.

3.5 Carbonate Alkalinity (CO_3^{2-}): Present when phenolphthalein alkalinity is not zero but is less than total alkalinity.

3.6 Bicarbonate Alkalinity (HCO_3^-): Present if phenolphthalein alkalinity is more than half the total alkalinity.

4.0 INTERFERENCES

4.1 Substances such as salts of weak organic and inorganic acids present in large amounts may cause interference in the electrometric pH measurements.

4.2 Precipitates, soaps, suspended solids, and oil and grease may also interfere by coating the pH electrode thus causing sluggish response.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

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5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 There are no specialized safety concerns associated with this method.

5.3 PRIMARY MATERIALS USED

5.3.1 There are no materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 pH meter that uses a combination electrode that can be read to 0.05 pH units.

6.2 Magnetic stirrer, pipettes, flasks and other standard laboratory equipment.

6.3 Burets: Class A 10 ml, 25 ml.

6.4 Beaker: Pyrex (Kimax) 50 ml, 150 ml or larger.

6.5 Ultrasonic bath or shaker, for soils only.

6.6 Analytical Balance.

6.7 Digestion Tube: 130mL

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI water obtained from the Millipore unit.

7.3 Alkalinity LCS/Spike Intermediate Standard:

7.3.1 $CaCO_3$ 1000 mg/L, place 1.0 ± 0.1 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and dilute to the mark with DI water. Good for 6 months.

7.4 Sulfuric Acid, concentrated.

7.4.1 Sulfuric acid titrant, 0.02N: Available commercially.

7.4.1.1 If sulfuric acid is not purchased pre-standardized prepare by diluting 1.2 ml concentrated sulfuric acid to two liters with DI water.

7.4.1.2 See section 10 for standardization.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Do not open sample container prior to analysis.

8.3 Store sample at 4˚C ± 2˚C.

8.4 Samples do not require chemical preservation.

9.0 QUALITY CONTROL

9.1 Batch

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a <u>method blank</u>, a <u>Laboratory Control Sample</u> (LCS), a Matrix Spike (MS) and a <u>Sample Duplicate</u> (DU). In the event that there is insufficient sample to analyze a duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 Method Blank

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water.

9.2.4 For Soil analyses, the method blank is comprised of boiling chips.

9.3 Laboratory Control Sample

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Calcium Carbonate.

9.3.4 For Soil analyses, the LCS is comprised of boiling chips fortified with Calcium Carbonate.

9.3.5 In addition to the standard LCS, a high level LCS (HLCS) is also performed for this test.

9.4 Matrix Spike (MS)

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 Sample Duplicate (DU)

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

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9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.6 Procedural Variations/ Nonconformance and Corrective Action

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibrate the pH meter with pH 4.0, 7.0 and 10.0 buffers, see SOP ST-WC-0011 for details.

10.2 Standardization of Acid (if needed):

10.2.1 Prepare sulfuric acid solution of approximate normality.

10.2.2 Standardize against 40 mL 0.05N Na_2CO_3 solution, with about 60 mL water in a beaker by titrating potentiometrically to pH of about 5.

10.2.3 Lift out electrodes rinse into the same beaker.

10.2.4 Boil gently for 3 to 5 minutes under a watch glass cover.

10.2.5 Cool to room temperature, rinse cover glass into beaker.

10.2.6 Finish titrating to the pH inflection point.

10.2.7 Record volume of titrant.

Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where:

A = g Na_2CO_3 weighed into 1 liter (Section 7.2)

B = ml Na_2CO_3 solution

C = ml acid used to inflection point

10.3 The low level LCS and high level LCS serve as calibration check.

11.0 PROCEDURE

11.1 Potentiometric Titration of Water Samples

11.1.1 Fill a 25 ml buret with 0.02N sulfuric acid titrant (7.1.4).

11.1.2 Place 50 ml of sample into a 100 ml digestion tube by pipetting with pipette tip near bottom of flask.

11.1.2.1 If testing low alkalinity use 200 ml of sample.

11.1.3 For the method blank use DI water in same amount as samples.

11.1.4 For the LCS and HLCS use 10 ml and 20 ml of 1000 mg/L $CaCO_3$ bring to 50 ml with DI water, respectively.

11.1.5 For MS, spike 5 mL of 1000 mg/L $CaCO_3$ bring to 50mL of sample

11.1.5.1 Report MS for total only.

11.1.6 Measure and record pH of sample.

11.1.6.1 If the pH > 8.3, titrate to pH 8.3 by adding titrant being careful to stir thoroughly, but gently, to allow display of pH meter to obtain equilibrium.

11.1.7 Record volume of titrant used.

11.1.8 Continue titrating down to pH 4.5.

11.1.9 Record the total volume of titrant used from starting point.

11.1.10 For low alkalinity samples (< 20 mg $CaCO_3$/L), using a 10 ml buret using 200mL of sample titrate to 4.5 and continue titrating down to pH 4.2. Record total number of ml from initial to pH 4.5 and from initial to pH 4.2.

11.1.10.1 The method blank is analyzed using the low alkalinity method.

11.2 Potentiometric Titration of Soil/Sediment Samples

11.2.1 Weigh 10 grams of sample into a 130 ml digestion tube. Add 100ml of DI water.

11.2.1.1 For the method blank use 10g glass beads. Add 100ml of DI water

11.2.1.2 For LCS use 10mL of 1000mg/L $CaCO_3$ to 100mL DI water with 10g glass beads.

11.2.2 Put sample on shaker table for 10 minutes.

11.2.3 Let sample settle.

11.2.4 Pipette 50ml of sample into digestion tube to be titrated.

11.2.5 Record volume of extract to be titrated.

11.2.6 Measure and record pH of sample.

11.2.6.1 If pH > 8.3 titrate down by adding titrant to pH 8.3 being careful to stir thoroughly, but gently, to allow display of pH meter to obtain equilibrium.

11.2.7 Record volume of titrant.

11.2.8 Continue titrating down to pH 4.5.

11.2.9 Record the total volume of titrant used from starting point.

11.2.10 For low alkalinity samples (< 200 µg $CaCO_3$/g), using a 10ml buret, continue titrating down to pH 4.2.

11.2.11 Record total number of ml from initial to pH 4.5 and from initial to pH 4.2.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 The titrant normality calculation is found in section 10.2 of this SOP.

12.3 Perform a percent solid determination on a separate soil sample aliquot if the calculation is on a dry weight basis.

12.4 Concentration calculations:

Water: Phenolphthalein alkalinity, mg/L $CaCO_3$

$$CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml sample}}$$

Soil/sediments: Phenolphthalein alkalinity, µg /g $CaCO_3$

$$CaCO_3 = \frac{A \times N \times 50{,}000 \times V}{TV \times W \times S}$$

where:

A	=	ml titrant used to reach pH 8.3
N	=	Normality of titrant.
TV	=	Titrated Extract Volume (L)
W	=	Weight of soil (g)
V	=	Extract Volume (L)
S	=	Solid fraction (% solids/100)

12.5 For Total Alkalinity concentrations:

Waters: Total alkalinity, mg/L $CaCO_3$

$$CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml sample}}$$

Soil/sediments: Total Alkalinity, µg /g $CaCO_3$)

$$CaCO_3 = \frac{A \times N \times 50{,}000 \times V}{TV \times W \times S}$$

where:
A = ml titrant used to reach pH 4.5. Others, as defined above.

12.6 Low alkalinity samples (< 20 mg/l for waters, < 200 µg /g soils):

Waters: Total Alkalinity, mg/l $CaCO_3$

$$CaCO_3 = \frac{(2B - C) \times N \times 50{,}000}{\text{ml sample}}$$

Soil/sediments: Total Alkalinity, µg /g $CaCO_3$

$$CaCO_3 = \frac{(2B - C) \times N \times 50{,}000 \times V}{TV \times W \times S}$$

Where:
B = ml titrant used to reach pH 4.5.
C = Total ml titrant to reach from initial sample pH to 4.2 pH. Others, as defined above.

12.7 Alkalinity Relationships:

Results of Titration	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Concentration as $CaCO_3$
P = 0	0	0	T
P < 1/2 T	0	2P	T – 2P
P = 1/2 T	0	2P	0
P > 1/2 T	2P – T	2 (T – P)	0
P = T	T	0	0

where:
P = Phenolphthalein alkalinity
T = Total alkalinity

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036

13.2 Method Blank
13.2.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.
13.2.1.1 Project specific requirements, if more stringent than the lab routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet.
13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
13.2.2.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the samples must be re-prepped/re-analyzed unless their concentration is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)
13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in the LIMS.
13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM, (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.3.2.3 RPD excursion for LCS/LCSD – If spike recoveries are acceptable, data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes.

13.4 Sample Duplicate/ Matrix Spike/Matrix Spike Duplicate (DU/MS/MSD)

13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in LIMS.

13.4.2 Corrective Action for RPD not meeting acceptance criteria:

13.4.2.1 RPD Duplicate excursion – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM describing the physical evidence; otherwise the sample should be re-prepped and re-analyzed.

13.4.2.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.2.1.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.4.2.2.1.2 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.4.2.2.1.3 If the excursion is due to physically evident matrix interference, the data is reported with an NCM.

13.4.2.2.1.4 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with and NCM.

13.4.2.2.1.5 When the MS/MSD recoveries and the %RPD are outside criteria, the batch is re-prepped and re-analyzed for the affected analytes.

13.5 Sample result evaluation

13.5.1 Dilutions

13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.5.1.1.1 An NCM is written to document the reason for the dilution.

13.5.1.1.2 If the dilution causes the Spike(s) to be diluted out an NCM is written to document the excursion.

13.5.2 Insufficient Sample

13.5.2.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 There are no special waste streams associated with this method.

17.0 REFERENCES

17.1 EPA Manual 600/4-79-020, Methods for Chemical analysis of Water and Wastes, Revised March 1983, Method 310.1.

17.2 Standard Methods for the examination of Water and Waste Water, 1997, Method 2320B - 2011.

17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs, current revisions:

17.5.1 ST-QA-0002, Standard and Reagent Preparation
17.5.2 ST-PM-0002, Sample Receipt and Chain of Custody
17.5.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
17.5.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.5.5 ST-QA-0016, IDL/MDL Determination
17.5.6 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.5.7 ST-WC-0011, Analysis of pH in Water
17.5.8 ST-IP-0004, Labware Preparation for Inorganic and Trace Metals Analysis

18.0 MODIFICATIONS TO REFERENCE METHODS

18.1 310.1 and SM 2320B

18.1.1 Modified method for solid matrices by performing DI water leaching prior to titration.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Updated section 11.0, regarding the replacement of flasks, with the use of specimen cups.

19.2 Rev. 13;

- 19.2.1 No Changes, Annual Review

19.3 Rev. 14:

- 19.3.1 Added digestion tubes to section 6.0.
- 19.3.2 Replaced the use of specimen cups with digestion tubes throughout section 11.0

19.4 Rev. 15: (07/26/2013)

- 19.4.1 Removed references to QuantIMS and Clouseau
- 19.4.2 Updated section 13
- 19.4.3 Updated section 15

19.5 Rev 16: (05/11/2015)

- 19.5.1 Changed acronym for sample duplicate to DU, to match LIMS
- 19.5.2 Added methods to section 18
- 19.5.3 Added HLCS to section 9.3
- 19.5.4 Corrected name of HLCS in section 11.1.4
- 19.5.5 Corrected amount spiked for MS in section 11.1.5

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Title: THE ANALYSIS OF ANIONS BY ION CHROMATOGRAPHY
[SW-846 9056A; EPA 300.0]

Approvals (Signature/Date):

5/30/14 Kristen Ely — Date Inorganics Department Manager	5/30/14 Michael Ridenhower — Date Health & Safety Manager / Coordinator
5-30-14 Tony L. Byrd — Date Quality Assurance Manager	6/2/14 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-WC-0028 Rev. 20

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the determination of fluoride, chloride, nitrite-N, bromide, iodide, nitrate-N, orthophosphate, sulfate, and chlorate ions in water and soil by ion chromatography. The SOP is applicable to drinking water, ground and surface waters, domestic and industrial wastewater, atmospheric precipitation samples, treatment process samples and also solid matrices.

1.2 This SOP is based on the following methods: EPA Method 300.0, SW 846 method 9056A.

1.3 When retention times indicate possible peak misidentification, anion identification should be supported by the addition of spike solutions covering the anions of interest (i.e., the method of known addition).

1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 A filtered aliquot of sample is injected into a stream of carbonate-bicarbonate eluant in an ion chromatographic system consisting of a guard column, separator column, suppressor column and conductivity detector. The guard and separator columns are packed with low-capacity, strongly basic anion exchange resin. Ions are separated based on their affinity for the exchange sites of the resin. The separated anions are directed onto a suppressor column that contains cation exchange resin in the hydrogen form. The suppressor column reduces the background conductivity of the eluant to a low or negligible level (weakly conductive carbonic acid) and converts the anions to their highly conductive acid forms. The separated anions in their acid form are measured by conductivity. They are identified on the basis of retention time as compared to known standards. Quantification is accomplished by measuring the area of the resultant peaks and comparing to a calibration curve generated from known standards.

3.0 DEFINITIONS

3.1 Method of known addition - The single standard addition technique involving addition of a known standard to a sample to confirm the identity of a tentatively identified analyte.

3.2 Retention Time - The interval measured from the point of sample injection to the point of maximum peak height or area.

3.3 Resolution - The ability of a column to separate constituents under specified test conditions.

3.4 Eluant - The ionic mobile phase used to transport the sample through the exchange column.

3.5 Regenerant - A solution that converts and maintains an active form of the suppressor.

3.6 Linear Check Standard – The high level standard of the curve is run quarterly to demonstrate the linear range of the instrument. The Linear Check Standard must be within +/- 10% of the true result.

3.7 The ICV satisfies the QCS requirements found in method 300.0.

3.8 The LCS satisfies the requirements of the LFB found in method 300.0.

3.9 The MS satisfies the requirements of the LFM found in method 300.0.

3.10 The CCV satisfies the requirements of the IPC found in method 300.0.

4.0 INTERFERENCES

4.1 Any substance that has a retention time coinciding with that of any anion to be determined will interfere. For example, relatively high concentrations of low molecular weight organic acids (e.g., acetate and formate) interfere with the determination of chloride and fluoride. A high concentration of any one ion also interferes with the resolution of others. Sample dilution overcomes much interference. To resolve uncertainties of identification, use the method of known additions.

4.2 Spurious peaks or elevated baseline may result from contaminants in reagent water, glassware, or sample processing apparatus. Because small sample volumes are used, scrupulously avoid contamination.

4.3 Water from the sample injection will cause a negative peak or dip in the chromatogram when it elutes because its conductivity is less than that of the suppressed eluant. This dip usually occurs before fluoride and chloride. Any peak of interest eluting near the water dip must be sufficiently resolved from the dip to be accurately quantified. Adjustment of sample background may be accomplished by diluting the sample with eluant if sample dilution is required prior to analysis. Add an equivalent of 1.0 ml of a prepared eluant concentrate (solution that is 100 times more concentrated than the eluant used for analysis) per 100 ml of sample. It is important to prepare a blank using reagent water and eluant (100:1) to compensate for any anionic impurities present.

4.4 Samples and reagents containing particulates require filtration through a 0.45 µm filter to avoid fouling or clogging the resin columns.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
- 5.2.1 None

5.3 PRIMARY MATERIALS USED
- 5.3.1 There are no materials used in accordance with this method which have serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6 [redacted]

6.1 Ion chromatography units
- 6.1.1 ICS2500 (Jack)
- 6.1.2 ICS1500 (Claire)
- 6.1.3 DX500 (Sawyer)

6.2 Computer, IBM XT or equivalent

6.3 Chromeleon Software v.6.8

6.4 Sample loop: 50 µl for routine anions; 1000µl for iodide.

6.5 Guard column, Dionex IonPac AG14 or AG9-HC (AG16 for iodide).

6.6 Anion Separator, Dionex IonPac AS14 or AS9-HC (AS16 for iodide).

6.7 Self refrigerating suppressor (SRS), Dionex

6.8 Conductivity detector, Dionex.

6.9 Autosampler (AS40 or equivalent), Dionex.

6.10 Sample Vials, 5 ml, Dionex .

6.11 Filtercaps (For 5 mL vials), Dionex.

6.12 50 mL plastic tubes

6.13 0.45 micron membrane filters

6.14 Pump, Dionex Gradient.

6.15 Interface, Dionex advanced.

6.16 Balance, analytical, calibrated.

6.17 Spatula

6.18 Shaker Table

6.19 Centrifuge

6.20 Pipettes, calibrated.

6.21 Volumetric Flasks, 100-ml and 2-L.

6.22 Glass beads

7.0 REAGENTS AND STANDARDS

7.1 All standard and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI water from Milli-Q system

7.3 Nitrogen gas (50psi), ultra high purity. (Nitrogen may be used as an alternative)

7.4 Sodium carbonate (0.35M)/Sodium bicarbonate (0.10M) eluant concentrate, commercially available.
7.4.1 Eluant solution: Pour 250 ml sodium carbonate/bicarbonate concentrate into reagent water and dilute to 25 liters. The resulting solution is 1.0 mM in NaHCO3 and 3.5 mM in Na2CO3.

7.5 35mM NaOH Eluant (Iodide and Perchlorate)
7.5.1 – Add 3.67ml 50%(w/w) NaOH to 2.0L of DI.

7.6 High Stock Standard for Calibration and CCVs (CAL HIGH)
7.6.1 Certified stock standard purchased commercially. Stability is 1 year.

Ion	mg/L

Fluoride	2.5
Chloride	5
Nitrite-N	0.4
Bromide	5
Nitrate-N	1.0
Orthophosphate	20
Sulfate	20

7.7 Low Stock Standard for Calibration (CAL LOW)

7.7.1 Certified stock standard purchased commercially. Stability is 1 year.

Ion	**mg/L**
Fluoride	2
Chloride	4
Nitrite-N	0.4
Bromide	5
Nitrate-N	0.4
Orthophosphate	10
Sulfate	10

7.8 Iodide Calibration Stock Standard (1000 ppm): Certified stock standard purchased commercially. Stability is 1 year.

7.8.1 Iodide calibration intermediate (100 ppm): Dilute 1.0 ml of the 1000 ppm calibration stock standard to 10 ml with reagent water. Stability is 30 days.

7.9 Potassium Chlorate Calibration Stock Standard (1000 ppm): Certified stock standard purchased commercially. Stability is 1 year.

7.9.1 Potassium chlorate calibration intermediate (100 ppm): Dilute 1 ml of the 1000 ppm calibration stock standard to 10 ml with reagent water. Stability is 30days.

7.10 Stock Standard for LCS and ICV:

7.10.1 Certified stock standard purchased commercially. Stability is 1 year. Must be from a different source than the standards used for calibration.

Ion	**mg/L**
Fluoride	2.5
Chloride	5
Nitrite-N	0.4
Bromide	5
Nitrate-N	1.0
Orthophosphate	20
Sulfate	20
Chlorate	20

7.11 Iodide Stock Standard for LCS and ICV (1000 ppm): Certified stock standard purchased commercially. Must be from a different source than the standard used for calibration. Purchase yearly.

7.11.1 Iodide LCS/ICV intermediate (100 ppm): Dilute 1.0 ml of the 1000 ppm LCS/ICV stock standard to 10 ml with reagent water. Stability is 30days.

7.12 Matrix Spike for Waters

7.12.1 Certified stock standard purchased commercially. Stability is 1 year.

Concentration in mg/L						
F-	Cl-	NO2- -N	Br-	NO3--N	PO43-	SO42-
100	100	5	100	20	200	200

7.13 Calibration Standards (all except iodide or chlorate):

7.13.1 Pipette the appropriate volumes of the CAL LOW stock (7.7.1) and CAL HIGH stock (7.6.1) into 5 ml vials. Dilute to volume with DI water. Prepared fresh daily.

Standard	**ml of Stock**	**Concentration in mg/L**						
		F-	Cl-	NO2-	Br-	NO3-	PO43-	SO42-
CAL LOW	0.25	0.1	0.20	0.02	0.25	0.02	0.5	0.5
CAL HIGH	0.50	0.25	0.50	0.04	0.50	0.10	2.0	2.0
CAL HIGH	1.0	0.5	1.0	0.08	1.0	0.20	4.0	4.0
CAL HIGH	2.5	1.25	2.5	0.20	2.5	0.50	10.0	10.0
CAL HIGH	Straight	2.5	5	0.4	5	1	20	20

7.14 Calibration Standards (Iodide):

7.14.1 Pipette the appropriate volumes of the 100 ppm calibration intermediate (7.8.1) and 1000 ppm calibration stock (7.8) into 10 ml vials. Dilute to 5 ml with DI water. Prepare fresh weekly.

Standard	**ml of Iodide Standard**	**Concentration in mg/L**
DI Water	5 ml	0
Intermediate	0.025	0.5
Intermediate	0.100	2.0
Stock	0.020	4.0
Stock	0.050	10.0
Stock	0.100	20.0

7.15 Calibration Standards (Potassium Chlorate):

7.15.1 Pipette the appropriate volumes of the 100 ppm calibration intermediate (7.9.1) and 1000 ppm calibration stock (7.9) into 5 ml vials. Dilute to volume with DI water. Prepare fresh weekly.

Standard	**ml of Chlorate Standard**	**Concentration in mg/L**
DI Water	5 ml	0
Intermediate	0.025	0.5
Intermediate	0.10	2.0
Stock	0.02	4.0
Stock	0.05	10.0
Stock	0.10	20.0

7.16 ICV/LCS

7.16.1 Take 2 ml of the LCS standard (7.6.1) and dilute to 5 ml. Prepare fresh daily.

Ion	**mg/L**
Fluoride	1.0
Chloride	2.0
Nitrite-N	0.16
Bromide	2.0
Nitrate-N	0.4
Orthophosphate	8.0

Chlorate	8.0
Sulfate	8.0

7.16.2 ICV (Iodide): Pipette 0.020 ml of the 1000 ppm ICV/LCS stock solution (7.11) into 5 ml of DI water for a final concentration of 4.0 mg/L.

7.16.3 LCS (Iodide): Pipette 0.020 ml of the 1000 ppm ICV/LCS stock solution (7.11) into 5 ml of DI water for a final concentration of 4.0 mg/L.

7.17 Matrix Spike

7.17.1 Pipette 0.1 ml of solution (7.13) into 5 ml of sample. Prepare fresh daily.

Final Concentration in mg/L						
F-	Cl-	NO2- -N	Br-	NO3--N	PO43-	SO42-
2	2	0.1	2	0.4	4	4

7.17.2 MS (Iodide): Pipette 0.020 ml of the 1000 ppm ICV/LCS stock solution (7.11) into 5 ml of sample for a final concentration of 4.0 mg/L.

7.17.3 MS (Chlorate): Pipette 0.02 ml of the 1000 ppm ICV/LCS stock solution (7.12.1) into 5 ml of DI water for a final concentration of 4.0 mg/L.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
All samples should be collected in polyethylene or glass amber bottles.

8.2 Sample preservation for Method 9056A requires samples be kept at ≤ 6° C.

8.3 Method 300.0 Water and soil samples preservation and holding times for the anions are as follows:

Analyte	**Preservation**	**Maximum Holding Time (from collection)**
Fluoride	None	28 days
Chloride	None	28 days
Nitrite-N	Cool, 4 °C	48 hours
Bromide	None	28 days
Nitrate-N	Cool, 4 °C	48 hours
Orthophosphate	Cool, 4 °C	48 hours
Sulfate	Cool, 4 °C	28 days
Chlorate	Cool, 4 oC	28 days
Iodide	Cool, 4 oC	28 days (waters) 28 days (soils)

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Method Blank, a Laboratory Control Sample (LCS), Sample Duplicate (SD), Matrix Spike (MS) and Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water.

9.2.4 For Soil analyses, the method blank is comprised of glass beads that are put through the DI leach procedure.

9.3 **Laboratory Control Sample**

9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Anions.

9.3.4 For Soil analyses, the LCS is comprised of glass beads and DI water fortified with anions and put through the DI leach procedure.

9.4 **Sample Duplicate (SD)**

9.4.1 A sample duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.4.2 For EPA 300.0 & 9056A: SD must be performed with every sample batch.

9.5 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.5.3 **For 9056A**: MS/MSD samples must be performed with every sample batch.

9.5.4 **For EPA 300.0**: MS must be performed every 10 samples or per batch whichever is shorter.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 External standard calibration is used.

10.2 Sample peak areas are compared to peak areas of the standards.

10.3 Initial Calibration

10.3.1 Prepare Anion standards at a minimum of five concentration levels. The low level standard should be at or below the reporting limit for each anion. The other standards define the working range of the detector.

10.3.1.1 50 ul of concentrated eluant is added to each calibration standard.

10.3.2 Frequency:

10.3.2.1 Generate an instrument calibration curve at a minimum weekly.

10.3.2.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, new Eluant Stock, any significant changes in instrument operating parameters, and major instrument maintenance.

10.3.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica corporate policy, "Calibration Curves", CA-Q-S-005.

10.3.4 **Linear calibration using a least squares regression**

10.3.4.1 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte unless specified as lower by client requirements

10.3.4.2 r (correlation coefficient) must be ≥ 0.995

10.3.4.3 The laboratory forces each anion through zero, unless otherwise specified.

10.4 **Initial Calibration Verification/Initial Calibration Blank** (ICV/ICB)

10.4.1 The initial calibration accuracy is verified by analyzing a second source standard (ICV).

10.4.2 ICV Frequency:

10.4.2.1 Perform with each initial calibration

10.4.3 ICV Criteria:

10.4.3.1 The ICV must fall within 10% of the true value for that solution.

10.4.4 ICB Frequency:

10.4.4.1 An ICB is analyzed immediately following the ICV to monitor system cleanliness.

10.4.5 ICB Criteria:

10.4.5.1 The ICB result must fall within +/- the RL from zero.

10.4.6 If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

10.4.6.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.5 **Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB)**

10.5.1 Calibration is monitored throughout the analytical run through the analysis of a known standard.

10.5.2 A CCV is the same source as the calibration

10.5.3 CCV Frequency:

10.5.3.1 Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a mid-level calibration standard.

10.5.4 CCV Criteria:

10.5.4.1 The CCV must fall within 10% of the true value for that solution.

10.5.4.2 If a CCV has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (ie initial calibration). Samples after the last acceptable CCV require re-analysis.

10.5.5 CCB Frequency:

10.5.5.1 A CCB is analyzed immediately following each CCV.

10.5.6 CCB Criteria:

10.5.6.1 The CCB result must fall within +/- RL from zero.

10.6 Retention Time

10.6.1 Establishing Retention Time Windows
10.6.1.1 Retention Time (RT) Centroid established for each anion by using the midpoint standard of the ICAL curve when ICAL is performed.
10.6.1.2 The RT window is set at 5% for all anions. No overlapping of anions is present with a 5% RT window.
10.6.1.3 The ICVs/CCVs must be within the established retention time windows.
10.6.1.4 If standard analyte falls outside the RT window, stop analysis. Recalibrate instrument and rerun samples from last acceptable ICV/CCV.

11.0 PROCEDURE

11.1 Sample Preparation
11.1.1 Water samples are analyzed neat. Pipette sample into a 5-ml auto-sampler vial.
11.1.1.1 Prepare MS/MSD: Fortify additional sample aliquots with MS standard.
11.1.2 Soil Sample Extraction
11.1.2.1 Weigh four grams of sample (± 0.04g) into a 50 ml plastic tube.
11.1.2.1.1 Record sample weight on DI leach sheet.
11.1.2.2 Add 40ml DI water
11.1.2.2.1 Record DI volume on DI leach sheet.
11.1.2.3 Shaker table for 15 minutes.
11.1.2.3.1 Record start and stop time on DI leach sheet.
11.1.2.4 Let leachate stand 15 minutes or centrifuge for 15 minutes.
11.1.2.5 Filter the resulting slurry before injecting using a 0.45 micron (µm) membrane type filter before injecting into a 5 ml sample vial.
11.1.2.5.1 Prepare MS/MSD: Fortify a sample aliquot with MS spike solution.
11.1.3 50 ul of concentrated eluant is added to all standards, QC samples and client samples.

11.2 ICS 2500 Ion Chromatographs Set Up
11.2.1 Allow the instrument to become stable before beginning calibration (minimum 30 minutes of warm-up is required.)
11.2.2 Check that the reagent (eluant, regenerant) bottles are properly filled.
11.2.3 Turn gases on (Nitrogen).
11.2.4 Load standards and samples into the auto-sampler as per example run summary (Table 1).

Table 1

Autocal 1R (blank) (Cal1)
Autocal 2R (first std) (Cal2)
Autocal 3R (second std) (Cal3)
Autocal 4R (third std) (Cal4)
Autocal 5R (fourth std) (Cal5)
Autocal 6R (fifth std) (Cal6)
ICV
ICB
Samples (to a max. of 10 including Blank and LCS)
CCV/CCB (every 10 samples)

11.2.5 Make sure the Chromeleon server is connected.
11.2.6 Open the Browser page by opening the software Chromeleon. Type the Run into the sequence menu. Arrow down to append new samples starting with the sequence from 11.2.2.1. When finished, Save.
11.2.7 Load the auto-sampler with sequence from 11.2.2.1 and push Hold to Run on auto-sampler.
11.2.8 To start up the instrument, go to – DHJ9Q141_local → ICS_2500_1 folder → open the conductivity Pump LC25. Click on the START UP icon which turns on the pump and detector. Let stabilize for at least 15 minutes. Start sequence by going to Batch on the menu → Edit → pull open the sequence file → Start, or Panel icon.

11.2.9 Refer to the ICS 2500 owner's manual for additional instructions.

11.3 The analysis will proceed automatically from this point.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the ST-QAM.

12.2 Analyte identifications and calculations for calibrations and sample concentrations are done internally by the Chromeleon computer software.

12.2.1 Analyst judgment may be used to identify peaks that are outside the retention time windows set, but such identifications are verified by matrix spike performance.

12.3 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.

12.3.1 A dilution should target the most concentrated analyte in the middle of the working range.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria:No target analytes may be present in the method blank above the reporting limit.

13.2.1.1 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 <u>Method Blank Contamination</u> – If the Method Blank concentration exceeds the applicable criteria the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank..

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). When samples are run for compliance with the drinking water regulations, the LCS recovery must be 90 – 110%.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 <u>LCS Spike Recovery excursion (high)</u> – Samples with results less than the RL may be reported with an NCM, unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 <u>LCS Spike Recovery excursion (low)</u> – the batch is re-prepped and re-analyzed for the affected analytes.

.

13.3.2.3 <u>RPD excursion for LCS/LCSD – If spike recoveries are acceptable ,data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes.</u>

13.4 Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/SD)

13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 <u>MS/MSD Spike Rec. excursion</u> may not necessarily warrant corrective action other than narration.

13.4.2.1.1 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.4.2.1.2 If the excursion is due to a physically evident matrix interference, the data is reported with an NCM.

13.4.2.1.3 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with and NCM.

13.4.2.1.4 When the MS/MSD recoveries and the %RPD are outside criteria, the batch is re-prepped and re-analyzed for the affected analytes.

13.4.2.2 % RPD excursion for MS/MSD - sample/sample duplicate – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM.; otherwise the sample should be re-prepped and re-analyzed

13.5 Sample result evaluation

13.5.1 Dilutions

13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.5.1.1.1 An NCM is written to document the reason for the dilution.

13.5.1.1.2 If the dilution causes the Spike(s) to be diluted out An NCM is written to document the excursion.

13.6 Insufficient Sample

13.6.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

13.7 Manual Integration

13.7.1 Identified compounds are reviewed for proper integration. Integrations are performed automatically by the data system. If necessary, manual integrations are performed and are documented by the analyst. Manual integrations are denoted on the raw data with a "modified by #*analyst name*#" and a stamp detailing the reason for the integration. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices for details.

14 [redacted]

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

14.5 Method 300.0 requires an MDL study be run every six months.

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If by a GM meter the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 EPA Manual 600/4-84-017, "The Determination of Inorganic Anions in Water by Ion Chromatography", Method 300.0. Revision 2.1, august 1993

17.2 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 9056A, Revision 1, 2007, "Determination of Inorganic Anions by Ion Chromatography"

17.3 ICS 2500 Ion Chromatograph Operation's Manual.

17.4 TestAmerica St. Louis Quality Assurance Manual (QAM), current revision.

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (ST-HS-0002).

17.6 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

17.7 Test America Policy CA-Q-S-002, Acceptable Manual Integration Practices

17.8 Associated SOPs

17.8.1 ST-QA-0002, Standard and Reagent Preparation

17.8.2 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes

17.8.3 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.8.4 ST-QA-0016, IDL/MDL Determination

17.8.5 ST-QA-0036, Non-conformance Memorandum (NCM) Process

17.8.6 ST-PM-0002, Sample Receipt and Chain of Custody
17.8.7 ST-IP-0004, Labware Preparation for Inorganic and Trace Metal Analysis
17.8.8 ST-WC-0005, Percent Solids Determination, current revision.
17.8.9 ST-QA-0040, Manual Integration Procedure

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 EPA 300.0 and 9056A require a mulit-point calibration consisting of 3 standards plus a blank. TestAmerica St. Louis uses 5 standards plus a blank to establish linearity.

18.2 EPA 300.0 states analyzing a LCR every 6 months in addition to establishing an initial calibration. TestAmerica St. Louis performs a multi-point calibration and does not report values outside the instrument's calibration range and thus an LCR is not applicable to the sample data.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated section 7.10 regarding stability of certified stock.
19.2 Added Chlorate to the list of certified stock standards in section 7.10.1 and removed separate single source for chlorate (formerly section 7.12).
19.3 Added eluant information to section 10.3 and 11.1.
19.4 01/18/2011, Rev 16, Removed use of 10mls of iodine.
19.5 Removed testing of a MSD sample from EPA method 300.0.
19.6 Revision 17;
- 19.6.1 Updated and identified instruments used for Ion Chromatography in section 6.0.
- 19.6.2 Removed the use of a sonicator and replaced it with a shaker table in section 6.0. Included the use of a centrifuge in that same section.
- 19.6.3 Removed the use of Nitrogen gas and designated it as an alternative option in section 7.0.
- 19.6.4 Updated the shelf life of calibration stock standards throughout section 7.0.
- 19.6.5 Added Chlorate to the list of LCS standards in section 7.16.
- 19.6.6 Removed section 7.16.3, 7.16.5, 7.18, 7.19, 10.6.1.2.1, 12.4 and 18.3, duplicate information.
- 19.6.7 Include a Sample Duplicate in section 9.1.3 and defined the purpose in section 9.4.
- 19.6.8 Updated instructions for forcing curves through zero in section 10.3.4.3.
- 19.6.9 Updated instructions for RT windows for each analyte in section 10.6.1.2.
- 19.6.10 Included the use of a centrifuge in the leaching process in section 11.1.
- 19.6.11 Added Manual Integration Procedure SOP to references in section 17.0.

19.7 Revision 18:
- 19.7.1 Updated section 10.6 regarding establishing retention time windows for initial calibrations and the process of calculating that retention time.

19.8 Revision 19:
- 19.8.1 Annual Review, No Changes.

19.9 Revision 20:
- 19.9.1 Removed ICV and LCS identical reference in section 3.0.
- 19.9.2 Updated equipment and supplies in section 6.0.
- 19.9.3 Gas used changed from Helium to nitrogen in Section 7 & 11
- 19.9.4 Updated information regarding Sample Duplicates for EPA 300 and 9056A in section 9.4.
- 19.9.5 Removed r^2 criteria from section 10.3.
- 19.9.6 Added Manual Integration information to Sections 13 & 17
- 19.9.7 Removed the use of Leach Log spreadsheet - no longer needed as information is now captured in the LIMs.
- 19.9.8 Removed references to "Clouseau" and "Quantims" replacing with "LIMS".
- 19.9.9 Updated Section 13, incorporating corrective actions for QC excursions.

19.10 Revision 21:
- 19.10.1 Added High Capacity columns to sections 6.5 and 6.6.
- 19.10.2 Changed stability of Cal High stock to 1 year in 7.10.1.
- 19.10.3 Removed sentence instructing dilution factors to be entered into run schedule in section 11.2.6. Not needed with TALS.

19.10.4 Updated section 10.6 to state that the retention time window for all anions is set at 5% and causes no overlapping of RTs.

19.10.5 Added EPA Method 300.0 requirement for MDL study every six months to section 14.

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Title: HEXAVALENT CHROMIUM (COLORIMETRIC) [SW-846 METHODS 7196A]

Approvals (Signature/Date):

Kristen Ely — Date 06/02/15 Inorganics Manager	Michael Ridenhower — Date 6/3/15 Health & Safety Manager / Coordinator
Marti Ward — Date 6-2-15 Quality Assurance Manager	Elaine Wild — Date 6/2/15 Laboratory Director

This SOP was previously identified as SOP No. ST-WC-0033 Rev. 18

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the analysis of hexavalent chromium [Cr(VI)] by spectrophotometry.

1.2 This method is applicable to the measurement of Cr(VI) in EP/TCLP characteristic extracts, ground water, and domestic and industrial wastes at concentrations above 10 µg/L. This method is also used to analyze the digestate from the alkaline soil digestion described in SOP ST-IP-0014.

1.3 This SOP is based on SW-846 Methods 7196A.

1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Cr(VI) is determined colorimetrically by reaction with diphenylcarbazide in acid solution.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 The chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent, however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause significant interference.

4.2 Iron in concentrations greater that 1 mg/L may produce a yellow color, but the ferric iron color is not strong and does not interfere with the readings if the absorbance is measured at the appropriate wavelength.

4.3 Sample alkaline digestion preparation interferences are given in SOP, ST-IP-0014.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	Mg/M^3 (TWA) as CrO_3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA - Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Spectrophotometer for use at 540 nm with a 1 cm or longer cell.
6.2 Pipette (0.1-1 mL), Pipette (1-10 mL),
6.3 Stir plate
6.4 Stir bars
6.5 pH meter
6.6 Digestion Vessel – 50 mL certified.

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Deionized Water, obtained from the Milli-Q unit.

7.3 Potassium dichromate stock solution 1000 mg/L (primary source): Dissolve 2.8297 g of dried potassium dichromate, $K_2Cr_2O_7$ (analytical reagent grade), in reagent water and dilute to 1 liter. Commercially prepared stock solutions may also be used. Good for 1 year.

7.4 Potassium dichromate stock solution 1000 mg/L (second source): Using a manufacturer or lot different than the primary standard, dissolve 2.8297 g of dried potassium dichromate, $K_2Cr_2O_7$

(analytical reagent grade), in reagent water and dilute to 1 liter. Commercially prepared stock solutions may also be used. Good for 1 year.

7.5 Sulfuric acid, 10% (v/v): Dilute 10 ml. of distilled reagent grade or spectrograde quality sulfuric acid, H_2SO_4, to 100 ml. with reagent water.

7.6 Diphenylcarbazide solution: Dissolve 250 mg. 1,5-diphenylcarbazide in 50 ml. acetone. Store in a brown bottle. Prepare daily.

7.7 Acetone (analytical reagent grade): Use only material that is received and stored in containers with non-metal or non-metal-lined caps.

7.8 Cr(VI) Intermediate standard 100 mg/L (primary source): Pipette 10.0 ml. of stock $K_2Cr_2O_7$ (7.3) into a 100 ml volumetric flask. Dilute to volume with DI water. Good for six months.

7.9 Cr(VI) Intermediate standard 100 mg/L (second source): Pipette 10.0 ml. of stock $K_2Cr_2O_7$ (7.4) into a 100 ml volumetric flask. Dilute to volume with DI water. Good for six months.

7.10 Cr(VI) Calibration standard 5 mg/L (primary source): Pipette 5.0 ml of the 100 mg/L Cr(VI) Intermediate standard (7.8) into a 100 ml volumetric flask. Dilute to volume with DI water. Prepare weekly.

7.11 Cr(VI) QC standard 5 mg/L (second source): Pipette 5.0 ml of the 100 mg/L Cr(VI) Intermediate standard (7.9) into a 100 ml volumetric flask. Dilute to volume with DI water. Prepare weekly.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Water samples must be analyzed within 24 hours of collection. See the soil alkaline digestion SOP, ST-IP-0014 for soil collection and holding times.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a <u>method blank (MB)</u>, a <u>Laboratory Control Sample</u> (LCS), and <u>Matrix Spike (MS)/ Matrix Spike Duplicate</u> (MSD) or a <u>Sample Duplicate</u> (DU). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the LIMS.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 A Matrix Spike/Matrix Spike Duplicate (or Sample Duplicate) must be run every ten samples for water samples. Soil samples have a matrix spike/matrix spike duplicate (or sample duplicate) per QC batch.

9.4.3 If Matrix Spike recovery is outside the control limits (85-115%) dilute the original sample aliquot 10X and re-spike at a level in the midrange of the curve. If this recovery is also outside the 85-115% limits an interference is present.

9.5 **Sample Duplicate (DU)**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision. Must be run every 10 samples.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036

10.0 CALIBRATION AND STANDARDIZATION

10.1 Standard Cr(VI) curve for waters, prepared daily when analysis is performed:

10.1.1 For each calibration and QC standard, start with 45 mL of DI water added to the digestate tube. Spike with the following amounts:

STANDARD	AMOUNT SPIKED (5.0 mg/L Calibration Standard)	AMOUNT SPIKED (5.0 mg/L QC Standard)
0 µg/L	---	---
10 µg/L	0.1 mL	---
20 µg/L	0.2 mL	---
50 µg/L	0.5 mL	---
100 µg/L	1.0 mL	---
LCS/CCV (50 µg/L)	0.5 mL	---
ICV (50 µg/L)	---	0.5 mL

10.1.2 To each standard:

10.1.2.1 Add 1.0 mL diphenylcarbazide solution.

10.1.2.2 Add 10% H_2SO_4 to give a pH of 2 ± 0.5 (4-5 drops)

10.1.2.3 Bring volume up to 50mL with DI water

10.1.2.4 Let all standards stand for 15 minutes before reading

10.2 Standard Cr(VI) curve for soils, prepared daily when analysis is performed:

10.2.1 For each calibration and QC standard, start with 45 mL of DI water added to the digestate tube. Spike with the following amounts:

STANDARD	AMOUNT SPIKED (5.0 mg/L Calibration Standard)	AMOUNT SPIKED (5.0 mg/L QC Standard)
0 µg/L	---	---
10 µg/L	0.1 mL	---
100 µg/L	1.0 mL	---
300 µg/L	3.0 mL	---
500 µg/L	5.0 mL	---
CCV (250 µg/L)	2.5 mL	---
ICV (250 µg/L)	---	2.5 mL

10.2.2 To each standard:

10.2.2.1 Add 1.0 mL diphenylcarbazide solution.

10.2.2.2 Add 10% H_2SO_4 to give a pH of 2 ± 0.5 (4-5 drops)

10.2.2.3 Bring volume up to 50mL with DI water

10.2.2.4 Let all standards stand for 15 minutes before reading

10.3 Spectrophotometer operation

10.3.1 Turn on power to spectrophotometer.

10.3.2 Await completion of SELF-TEST.

10.3.3 Press up or down arrows to analytical wavelength 540nm.

10.3.4 Allow 30-minute lamp warm-up.

10.3.5 Open sample compartment door; insert zero point of curve into sample holder, close sample compartment door and press zero.

10.3.6 Fill cuvette with all standards and read absorbances.

10.4 The initial calibration consists of 4 points and a blank.

10.4.1 Calculate a linear regression equation using the corrected standard concentration versus the µg of Cr(VI) recorded.

10.4.2 The correlation coefficient for the calibration curve must be ≥ 0.995. If it is not, a new calibration curve must be prepared prior to the analysis of samples.

10.4.3 The Y-intercept must be less than +/- the reporting limit for the analyte. When specifically requested, less than 3 times the MDL is required.

10.5 Analyze an ICV/ICB immediately after the last calibration standard is analyzed.

10.5.1 Acceptance criteria for ICV: 85%-115%.

10.5.2 Acceptance criteria for ICB: < RL

10.5.3 When the measurement exceeds the control limits for the ICB or ICV, the analysis must be terminated, the calibration curve rerun, and the calibration reverified.

10.6 Analyze a CCV/CCB every 10 samples and at the end of the analytical sequence.

10.6.1 Acceptance criteria for CCV: 85%-115%.

10.6.2 Acceptance criteria for CCB: < RL
10.6.3 If a CCV is outside control limits, the analysis must be stopped. Correct the problem and rerun all samples analyzed after the last acceptable CCV.
10.6.4 If a CCB concentration is above the detection limit, rerun all samples analyzed after the last acceptable CCB.

11.0 PROCEDURE

11.1 WATER;

11.1.1 Prepare sample

11.1.1.1 Transfer 45 ml of each extract or water sample to a 50 ml certified digestion vessel.
11.1.1.2 Add 0.5 mL of Calibration Intermediate (5.0 mg/L) to MS/MSD.
11.1.1.3 Add 1 ml of the diphenylcarbazide solution to the digestion vessel for each sample and QC sample.
11.1.1.4 Add a few drops of 10% H_2SO_4 to the sample to give a pH of 2 ± 0.5.
11.1.1.5 Fill to volume (50ml) with sample.
11.1.1.6 Let stand for 15 minutes
11.1.1.7 Read spectrophotometrically at 540 nm.

11.1.1.7.1 NOTE: It is very important that each sample and standard is read at exactly the same period of time after adding the diphenylcarbazide.

11.1.2 Prepare a turbidity blank for each sample

11.1.2.1 Transfer 45 ml of each extract or water sample to a 50 ml certified digestion vessel.
11.1.2.2 Add a few drops of 10% H_2SO_4 to the sample to give a pH of 2 ± 0.5.
11.1.2.3 Fill to volume (50 mL) with sample
11.1.2.4 Let stand for 15 minutes.
11.1.2.5 Read spectrophotometrically at 540 nm.
11.1.2.6 Record the turbidity blank for each sample, method blank, and LCS. If the absorbance reading of the turbidity blank is above the calibration curve, the sample must be either (re)filtered or diluted until it is within the calibration curve.

11.1.3 Correct the absorbance by subtracting the absorbance of the turbidity blank from the total absorbance.

11.1.4 Record the sample ID, dilution (if any), and absorbance reading in LIMS

11.1.5 Sequence should be:

11.1.5.1 Calibration Standards
11.1.5.2 ICV
11.1.5.3 ICB
11.1.5.4 BLK
11.1.5.5 LCS
11.1.5.6 8 samples
11.1.5.7 CCV
11.1.5.8 CCB
11.1.5.9 10 samples
11.1.5.10 CCV
11.1.5.11 CCB

11.1.6 Repeat 10 sample CCV/CCB sequence as needed.

11.2 SOILS

11.2.1 For the digestion of soils, refer to SOP ST-IP-0014, "Alkaline Digestion for Hexavalent Chromium".

11.2.2 Prepare digestate:

11.2.2.1 Take a 20mL pre-portioned aliquot of the sample to be analyzed and with constant stirring with a stir bar, slowly add 5.0 M nitric acid solution to the beaker dropwise. Adjust the pH of the solution to 7.5 +/- 0.5 and monitor the pH with a pH meter. Document exact pH in LIMS

11.2.2.2 Add 0.5mL of Diphenylcarbazide solution (Color Indicator) for every 25mL of sample being analyzed.

11.2.2.3 Add a few drops of 10% H2SO4 to sample to reduce pH.

11.2.2.4 When sample reaches a pH of 3, start timer for 10 minutes.

11.2.2.5 Bring sample to a final pH of 2 ± 0.5 Document exact pH in LIMS

11.2.2.6 Bring sample to final volume of 25mL with DI water and place it on stir plate.

11.2.3 Prepare turbidity blank:

11.2.3.1 Take a 20mL pre-portioned aliquot of the sample to be analyzed and with constant stirring with a stir bar, slowly add 5.0 M nitric acid solution to the beaker dropwise. Adjust the pH of the solution to 7.5 +/- 0.5 and monitor the pH with a pH meter. Document exact pH in LIMS

11.2.3.2 Add a few drops of 10% H2SO4 to sample to reduce pH.

11.2.3.3 When sample reaches a pH of 3, start timer for 10 minutes.

11.2.3.4 Bring sample to a final pH of 2 ± 0.5 Document exact pH in LIMS

11.2.3.5 Bring sample to final volume of 25mL with DI water and place it on stir plate.

11.2.4 Caution: CO2 will be evolved. This step should be preformed in a well ventilated area.

11.2.5 Read spectrophotmetrically at 540nm.

11.2.5.1 NOTE: It is very important that each sample and standard is read at exactly the same period of time after adding the diphenycarbazide.

11.2.6 Correct the absorbance by subtracting the absorbance of the turbidity blank from the total absorbance.

11.2.7 Record the sample ID, weight of soil extracted, dilution, if any and absorbance reading in LIMS

11.2.12 Sequence is the same as noted in section 11.1.9.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the St. Louis ST-QAM.

12.2 The correlation coefficient is obtained from the concentration of the standards (µg/L) and the absorbance reading of the standards used to prepare the calibration curve. The sample results are calculated from the regression coefficient using the least squares fit.

12.3 Water concentrations Cr (VI), µg/L:

$$\text{Cr(VI), } \mu g/L = A \times B$$

where:
A = µg/L calculated from the standard curve (12.1)
B = dilution factor

12.4 Soil concentration Cr(VI), µg/g:

$$\text{Cr(VI), } \mu g/g = \frac{A}{W} x V x B$$

where:
A = µg/L calculated from the standard curve (12.1)
B = dilution factor
W = Sample weight in grams
V = Extraction volume in liters

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.1 Method Blank

13.1.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.

13.1.1.1 Project specific requirements, if more stringent than the lab routine procedure (e.g. no target analytes present above ½ RL), will be noted in LIMS

13.1.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.1.2.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the samples must be re-prepped/re-analyzed unless their concentration is less than the RL or greater than ten times the concentration found in the blank.

13.2 Laboratory Control Sample (LCS)

13.2.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in the LIMS.

13.2.2 Corrective Action for LCS not meeting acceptance criteria:

13.2.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM, (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.2.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

1.1.1.1. .

13.2.2.3 RPD excursion for LCS/LCSD – If spike recoveries are acceptable, data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes.

13.3 Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/DU)

13.3.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in LIMS.

13.3.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.3.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.3.2.1.1 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.3.2.1.2 If the excursion is due to physically evident matrix interference, the data is reported with an NCM.

13.3.2.1.3 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with and NCM.

13.3.2.1.4 When the MS/MSD recoveries and the %RPD are outside criteria, the batch is re-prepped and re-analyzed for the affected analytes.

13.3.2.2 % RPD excursion for MS/MSD - sample/sample duplicate – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM describing the physical evidence; otherwise the sample should be re-prepped and re-analyzed

13.4 Sample result evaluation

13.4.1 Dilutions

13.4.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.4.1.1.1 An NCM is written to document the reason for the dilution.

13.4.1.1.2 If the dilution causes the Spike(s) to be diluted out an NCM is written to document the excursion.

13.5 Insufficient Sample

13.1.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", 3rd Edition, Method 7196A, Final Update III.

17.2 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", 3rd Edition, Method 3060A, Final Update III.

17.3 Instruction manual for Model 501/601 Milton Roy Spectrophotometer.

17.4 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.6 Associated SOPs:

17.6.1 ST-IP-0004, "Glassware Preparation for Inorganic and Trace Metal Analysis", current revision.
17.6.2 ST-IP-0014, "Alkaline Digestion for Hexavalent Chromium", current revision.
17.6.3 ST-PM-0002, Sample receipt and Chain of Custody
17.6.4 ST-QA-0002, "Standards Preparation", current revision.
17.6.5 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.6.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.6.7 ST-QA-0016, IDL/MDL Determination
17.6.8 ST-QA-0036, "Non-conformance Memorandum (NCM) Process", current revision.

18.0 CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHODS

18.1 Method 7196A states that the lab should, for verification, "analyze a second 10 mL aliquot of the pH adjusted filtrate that has been spiked with Cr(VI). The amount of the spike should be double the concentration found in the original aliquot." TestAmerica St. Louis spikes the aliquot with a spike level in the mid-range of the curve.

18.2 TestAmerica St. Louis does not perform an alternative method if interference persists after sample dilution.

18.3 TestAmerica St. Louis runs a second source ICV at the beginning of the run in place of a second source CCV every 15 samples. A calibration verification (CCV) is run every ten samples.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated section 9.0 regarding Matrix Spike (MS) and Matrix Spike Duplicate (MSD) sample runs.

19.2 Revision 13;

19.2.1 Updated section 6.0 with new equipment and supplies.
19.2.2 Updated the number of points needed for an initial calibration in section 10.4.
19.2.3 Updated the reporting of the Y-intercept to be +/-the reporting limit in section 10.4.3

19.2.4 Updated spreadsheets to include y intercept calculation.

19.3 Revision 14:

19.3.1 Updated the attachment 1 spreadsheet to include a column for the pH of the adjusted digestate.

19.3.2 Updated procedures in section 11.0 regarding the amount of sample/extract used for water analysis.

19.3.3 Added directions for preparing a turbidity blank in section 11.4.

19.4 Revision 15:

19.4.1 Updated section 9.5, added requirement for sample duplicate to be run every ten samples.

19.4.2 Added specific pH criteria to Spreadsheet 1

19.5 Revision 16:

19.5.1 Updated sample pH requirements in section 11.2.6. Also added that this final pH should be documented on spreadsheet.

19.6 Revision 17:

19.6.1 In section 11.1.4 added 1mL of acetone to be used to prepare turbidity blanks.

19.7 Revision 18: (07/30/2013)

19.7.1 Removal of references to QuantIMS and Clouseau.

19.7.2 Clarified sections 7.3 and 7.4 regarding primary and second source standards, their concentrations, and expirations.

19.7.3 Clarified sections 7.8 and 7.9 regarding primary and second source standards and their concentrations.

19.7.4 Added sections 7.10 and 7.11 to reflect the laboratory's use of 5 mg/L intermediate stocks.

19.7.5 Updated sections 10.1 and 10.2 to clarify which intermediate stocks are used in making the calibration curves, ICVs, and CCVs.

19.7.6 Updated section 10.5.1 to clarify that limits are for ICV.

19.7.7 Added section 10.5.2 to state limit for ICB.

19.7.8 Updated section 10.6.1 to clarify that limits are for CCV.

19.7.9 Added section 10.6.2 to state limit for CCB.

19.7.10 Section 13 updated with corrective actions

19.7.11 Updated Spreadsheet 1 & 2 attachments

19.8 Revision 19: (xx/xx/2015)

19.8.1 Re-wrote section 10 and 11 for clarification purposes

19.8.2 Changed name of 5 mg/L intermediates in section 7

19.8.3 Extended expiration date for the intermediate standard to six months in Section 7.

19.8.4 Changed acronym for sample duplicate to match LIMS

19.8.5 Removed references to spreadsheets and client requirement sheets, changed to reference LIMS throughout SOP.

19.8.6 Removed copies of spreadsheets that are no longer used

19.8.7 Added directions for turbidity blank for soils (section 11.2)

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Title: DETERMINATION OF SOLIDS IN WATER AND WASTES [EPA 160; Standard Method 2540]

Approvals (Signature/Date):

05/29/15	5/29/15
For Kristen Ely Inorganics Manager Date	Michael Ridenhower Health & Safety Manager / Coordinator Date
FOR 05-29-2015	5/29/15
Marti Ward Quality Assurance Manager Date	Elaine Wild Laboratory Director Date

This SOP was previously identified as SOP No. ST-WC-0036 Rev. 12

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the determination of total solids, total suspended solids, total dissolved solids, volatile solids, volatile dissolved solids, and volatile suspended solids using gravimetric techniques. This SOP is based on the residue methods contained in Methods for Chemical Analysis of Waters and Wastes (MCAWW) and Standard Methods for the Examination of Water and Wastewater (SM).

1.2 This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.3 The Total Solids protocol is based on MCAWW Method 160.3 and SM 2540B.

1.4 The Total Dissolved Solids protocol is based on MCAWW Method 160.1 and SM 2540C.

1.5 The Total Suspended Solids protocol is based on MCAWW Method 160.2 and SM 2540D.

1.6 The Volatile Solids protocol is based on MCAWW Method 160.4 and SM 2540E.

1.7 The Volatile Dissolved Solids protocol is based on MCAWW Method 160.4 and SM 2540E.

1.8 The Volatile Suspended Solids protocol is based on MCAWW Method 160.4 and SM 2540E.

1.9 The Settleable Solids protocol is based on method MCAWW Method 160.5 and SM 2540F.

1.10 The methods cover a practical range of 10 mg/L to 20,000 mg/L (TSS: 4 mg/L - 20,000 mg/L). The final residue weight must not exceed 200 mg.

2.0 SUMMARY OF METHOD

2.1 Total Solids (TS): A well-mixed aliquot of the sample is quantitatively transferred to a preweighed evaporating dish and evaporated to dryness at 103-105 °C. The increase in weight over that of the empty dish represents the total solids.

2.2 Total Dissolved Solids (TDS): A well-mixed sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a preweighed evaporating dish and is evaporated to dryness and then dried to constant weight at 180 °C. The increase in weight over that of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS/VSS determination.

2.3 Total Suspended Solids (TSS): A well-mixed sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to constant weight at 103-105 °C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination. The filter from this procedure may also be used for VSS analysis.

2.4 Volatile Solids (VS): The residue obtained from the determination of total solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile solids.

2.5 Volatile Dissolved Solids (VDS): The residue obtained from the determination of total dissolved solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile dissolved solids.

2.6 Volatile Suspended Solids (VSS): A well-mixed sample is filtered through a glass fiber filter to separate the suspended material. The filter is dried and weighed, then ignited at 550 °C and reweighed. Volatile suspended solids are determined from the weight loss after ignition. The filter from the analysis of TSS may be used for the determination of VSS, if manufacturer states that it is acceptable for volatile solids.

2.7 Settleable matter is measured volumetrically with an Imhoff cone.

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3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Total Solids (TS): The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 103-105 °C. Total solids include "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

3.3 Total Dissolved Solids (TDS): Those solids capable of passing through a glass fiber filter and dried to constant weight at 180 °C. TDS is also referred to as filterable residue.

3.4 Total Suspended Solids (TSS): Those solids which are retained by a glass fiber filter and dried to constant weight at 103-105 °C. TSS is also referred to as non-filterable residue.

3.5 Volatile Solids (VS): The portion of total solids which is lost on ignition at 550 °C.

3.6 Volatile Dissolved Solids (VDS): The portion of total dissolved solids which are lost on ignition at 550 °C.

3.7 Volatile Suspended Solids (VSS): The portion of suspended solids which is lost on ignition at 550 °C.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2 Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted in an NCM.

4.4 Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter.

4.5 Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, should be included in the sample and dispersed by a blending device before taking an aliquot.

4.6 Filtration apparatus filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7 The temperature at which the residue is dried has an important bearing on the results because weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8 Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

4.9 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

4.10 Samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.11 Too much residue in the drying vessel will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

4.12 Some samples may have fine suspended solids which will pass through the glass fiber filter causing high TDS results.

4.13 Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.14 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

4.15 The volatile solids tests are subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample.

4.16 For some samples, a separation of settleable and floating material will occur; in such cases the floating materials are not measured. (If client requires floating matter be included, TSS should be run.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 Oily samples or those that contain volatile chemicals may ignite during this procedure. In the case of a fire, the muffle should be turned off and allowed to cool before the sample can be removed and put under a hood.

5.2.2 Extreme care should be used when using a muffle furnace, as temperatures are extremely high. Gloves designed for dealing with extreme temperatures must be used when handling any samples or glassware that has been in a muffle furnace.

5.3 PRIMARY MATERIALS USED

5.3.1 There are no materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical balance capable of weighing to 0.0001 g.

6.2 Vacuum filtration apparatus.

6.3 Vacuum pump equipped with moisture trap.

6.4 Glass fiber filter disks, 47 mm, without organic binder (Gelman Type A/E) or equivalent.
Note: Commercially available pre-weighed filters may be used for TSS and VSS analyses.

6.5 Evaporating dishes, 100 mL, made of porcelain, high-silica glass. These must be thoroughly cleaned, rinsed with deionized water, and baked at 180 ± 2 °C for at least one hour before use. Store in a desiccator.

6.6 Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes.

6.7 Desiccant containing a color indicator of moisture concentration.

6.8 Drying ovens set at 103-105 °C and 180 ± 2 °C. Separate ovens should be maintained at appropriate temperatures if possible.

6.9 Muffle furnace (550 °C).

6.10 Thermometers, NIST traceable.

6.11 Conductivity meter

6.12 Class A Graduated cylinders, "to contain," assorted sizes.

6.13 Volumetric pipettes, wide-bore, Class A, assorted sizes.

6.14 Aluminum weighing dishes large enough to hold a 47 mm filter.

6.15 Forceps for handling filters.

6.16 Crucible tongs.

6.17 Gloves capable of providing protection at 550 °C.

6.18 Imhoff cones

6.19 Magnetic stir bars and stir plate

6.20 Glass stir rod

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards preparation, documentation and labeling must follow the requirements of ST-QA-0002, current revision.

7.2 DI water obtained from Milli-Q System unit

7.3 LCS solution (500 mg/L TSS and TDS, 1000 mg/L TS): Place 500.0 mg of diatomaceous earth (infusorial earth or celite 545) and 500 mg sodium chloride into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months.
Note: Commercially available reference materials may also be used.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples should be collected in either plastic or glass bottles.

8.3 Samples must be stored at 4 °C ± 2 °C to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

8.4 Holding time: samples are to be analyzed within 7 days of collection for all solids analyses.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a <u>method blank</u>, a <u>Laboratory Control Sample</u> (LCS), and <u>Sample Duplicate</u> (DU).

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 The method blank is comprised of DI water.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch for TSS, TDS and TS. There is no applicable LCS for VSS, VDS and VS.

9.3.3 Total Solids combines 500mg/L of Celite and 500 mg/L of NaCl for a true value of 1000 mg/L

9.3.4 For TSS, the LCS is 500 mg/L of Celite.

9.3.5 For TDS, the LCS is 500 mg/L of NaCl.

9.4 **Sample Duplicate**

9.4.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5 **Procedural Variations/ Nonconformance and Corrective Action**

9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and pipette calibrations must be checked daily when used. Refer to SOP ST-QA-0005

10.1.1 The lower calibration weight must be a maximum of 200 mg.

10.2 Oven temperature must be checked daily and recorded in the oven temperature logbook and in LIMS.

10.3 Conductivity of the water must be monitored and recorded in the Conductivity Logbook daily. The maximum permissible conductivity is 1.0 umhos/cm (at 25 °C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

11.0 PROCEDURE

11.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted in an NCM.

11.2 If samples are visibly oily, this should be noted in an NCM.

11.3 If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted in an NCM and reporting limits must be adjusted appropriately.

11.4 Proceed to the appropriate section for the desired method:

11.5 <u>Total Solids</u>

11.5.1 Preparation of Evaporating Dish

11.5.1.1 If only total solids are to be measured, heat a clean dish to 103-105 °C for one hour. If volatile solids are to be measured in addition to total solids, heat the clean evaporating dish at 550 °C for one hour in a muffle furnace.

11.5.1.2 Remove the dish from the muffle furnace using tongs and heat resistant gloves.

11.5.1.3 Cool and store dish in desiccator until dish reaches room temperature or until needed.

11.5.1.4 Weigh the dish immediately before use to the nearest 0.1 mg. Record the weight in LIMS.

11.5.2 Sample Analysis

11.5.2.1 Add stir bar to sample container and place on stir plate.

11.5.2.2 Pipet 100 mL of the well mixed sample to a pre-weighed, labeled dish. Record the volume of sample (to the nearest mL) in LIMS.

11.5.2.2.1 Choose an aliquot of sample (usually 100mls) sufficient to contain a residue of at least 10 mg but less than 200 mg. To obtain a weighable residue, successive aliquots of sample may be added to the same dish after evaporation.

11.5.2.2.2 If the sample is known to contain > 2000 mg/L dissolved solids, it should be diluted. Prescreening may be performed using a conductivity meter or tester to determine the required sample volume or dilution. For this purpose, the following formula should be used to approximate the required sample volume:
(2000 x 100) / Conductivity = mL sample to use

11.5.2.2.3 For the LCS, measure 100 mL of the LCS Solution and pour into the dish.

11.5.2.2.4 For the MB, measure 100 mL of DI water and pour into the dish.

11.5.2.3 Evaporate the sample to dryness on a steam bath or in a drying oven. If evaporation is performed in a drying oven, the temperature should be lowered as necessary (to

approximately 2 degrees below boiling) to prevent boiling and splattering of the sample.

11.5.2.4 Dry the evaporated sample for at least one hour at 103-105 °C.

11.5.2.5 Record the date, time, and oven temperature in LIMS when the samples are initially placed in the oven and again when they are removed from the oven.

11.5.2.6 Using tongs or gloves, remove the weighing dish from the oven and place in a desiccator and cool to room temperature, a minimum of two hours.

11.5.2.7 Weigh the dish to the nearest 0.1 mg. Record the weight in LIMS.

11.5.2.8 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained. Constant weight is demonstrated by weight loss less than or equal to 0.5 mg. If more than three cycles are required, write a NCM.

NOTE: When weighing dried sample, be alert to any change in weight due to air exposure and/or sample degradation.

11.5.2.9 If volatile solids are to be determined, treat the residue according to Section 11.8.

11.5.2.10 Calculate results according to the equation provided in Section 12. Use the final weight achieved for calculating TS.

11.6 Total Dissolved Solids

11.6.1 Preparation of Evaporating Dishes

11.6.1.1 If only total dissolved solids are to be measured, heat clean dish to 180 ± 2 °C for one hour. If volatile dissolved solids are to be measured in addition to TDS, heat the clean evaporating dish at 550°C for one hour in a muffle furnace.

11.6.1.2 Heat resistant gloves and tongs must be used when removing items from the muffle furnace.

11.6.1.3 Store and cool dish in desiccator until dish reaches room temperature or until needed, a minimum of two hours.

NOTE: Always transfer the dish with gloves or tongs to prevent added weight due to oil from fingerprints.

11.6.1.4 Weigh the dish immediately before use to the nearest 0.1 mg. Record the weight of the dish in LIMS.

11.6.2 Sample Analysis

11.6.2.1 Thoroughly rinse the entire filtration apparatus with DI water before filtering each sample.

11.6.2.2 Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using DI water, and begin suction.

NOTE: If the sample also requires TSS, preweigh the prepared filter and refer to Section 11.7 for additional guidance.

11.6.2.3 Add stir bar to sample container and place on stir plate.

11.6.2.4 Pipet 100 mL (or a smaller portion which will yield between 10 and 200 mg dried residue) sample to the funnel. If the Total Dissolved Solids content is low, a larger volume may be filtered. If more than 10 minutes are required to complete filtration, decrease sample size.

NOTE: Multiple filters may be used if performing only TDS analysis.

11.6.2.4.1 Record the volume of sample used (to the nearest mL) in LIMS.

11.6.2.4.2 For the method blank, process 100 mL of DI water as the sample.

11.6.2.5 Filter the sample through the glass fiber filter.

11.6.2.6 Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of DI water and allow for complete drainage between washings. Continue to apply vacuum for about three minutes after filtration is complete to remove as much water as possible.

11.6.2.7 Transfer the filtrate (including the washings) to a pre-weighed evaporating dish. Rinse the receiving flask with 10-25 mL of DI water and transfer washings into the dish to ensure complete transfer of the sample.

11.6.2.8 Evaporate the samples to dryness on a steam bath or in an oven set slightly below boiling. If filtrate volume exceeds dish capacity, add successive portions to the same dish after evaporation.

11.6.2.9 Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

11.6.2.10 Record the date, time, and oven temperature in LIMS when the samples are initially placed in the oven and again when they are removed from the oven.

11.6.2.11 Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.6.2.12 Weigh the dish to the nearest 0.1 mg. Record the combined weight of the dried residue and the dish in LIMS.

11.6.2.13 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained or weight loss is less than 0.5 mg. If more than three cycles are required, write a NCM.

11.6.2.14 Calculate results according to the equation in Section 12. Use the final weight achieved for calculating TDS.

11.7 Total Suspended Solids

11.7.1 Preparation of Glass Fiber Filter Disc

NOTE: As an alternative pre-weighed filters may be purchased for use under this method.

11.7.1.1 Place the glass fiber filter discs, one at a time, on the membrane filter apparatus or insert into bottom of a suitable crucible with wrinkled surface up.

11.7.1.2 While vacuum is applied, wash the disc with three successive 20 mL volumes of DI water.

11.7.1.3 Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.

11.7.1.4 Remove filter from membrane filter apparatus and place in a labeled, aluminum weighing dish (or remove both the crucible and filter if a crucible is used), and dry in an oven at 103-105 °C for one hour.

NOTE: If volatile suspended solids (VSS) are to be determined as well as total suspended solids, the filter must be heated at 550 ^{o}C for 15 minutes after analyzing for TSS. The VSS filter must be specifically recommended by the manufacturer.

11.7.1.5 Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature, a minimum of one hour.

11.7.1.6 Weigh the cooled filter using an analytical balance to the nearest 0.1 mg. Handle the filters or crucible/filter only with forceps. Record the weight and the dish identification number in LIMS.

11.7.1.7 Repeat the drying, cooling, and weighing cycle until a constant weight is obtained or weight loss is less than 0.5 mg.

11.7.2 Selection of Sample Volume

11.7.2.1 For a 47 mm diameter filter, filter *100* mL of sample.

11.7.2.1.1 Note a larger filter, 90mm, may be used if the sample is suspected to contain a high amount of solid material.

11.7.2.2 If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

NOTE: If the sample appears high in TSS, start with a sample volume < 100 mL.

11.7.2.3 For the method blank, process 100 ml of DI water as a sample.

11.7.2.4 For the LCS, process 100 ml. of the LCS solution as a sample.

11.7.3 Sample Analysis

11.7.3.1 Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using DI water and begin suction.

NOTE: Handle the filters or crucible/filter only with forceps.

11.7.3.2 Add stir bar to sample container and place on stir plate.

11.7.3.3 Pipet 100 mL (or a smaller portion which will yield between 10 and 200 mg dried residue) sample to the funnel.

11.7.3.3.1 A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Filter 25 mL at a time until filtration slows. Record the volume of sample filtered (to the nearest mL) in LIMS.
NOTE: If Total Dissolved Solids (TDS) is also required, the filtrate may be used.

11.7.3.4 With suction on, rinse the filter, suspended solids residue, and filter funnel wall with three 10 mL portions of DI water allowing complete drainage between washings.

11.7.3.5 Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

11.7.3.6 Carefully remove the filter from the filter support and transfer to an aluminum weighing dish. Alternatively, remove the crucible and filter from the crucible adapter.

11.7.3.7 Dry the filter at least one hour at 103-105 °C.

11.7.3.8 Record the date, time, and oven temperature in LIMS when the samples are initially placed in the oven and again when they are removed from the oven.

11.7.3.9 Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.7.3.10 Weigh the dish (to the nearest 0.1 mg), and record the weight in LIMS.

11.7.3.11 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 0.5 mg. If more than three cycles are required, write a NCM.

11.7.3.12 The final residue weight must not exceed 200 mg.

11.7.3.13 If volatile suspended solids are to be determined, proceed to the Volatiles Suspended Solids section.

11.7.3.14 Calculate the results using the formula given in Section 12. Use the final weight achieved for calculating TSS.

11.8 Volatile Solids

11.8.1 Heat muffle furnace up to temperature (550 °C).

11.8.2 Weigh dish and residue generated by Total Solids test.

11.8.3 Place evaporating dish containing residue generated by Total Solids section in muffle furnace to ignite the residue.

11.8.4 Record the date, time, and oven temperature in LIMS when the samples are initially placed in the oven and again when they are removed from the oven.

11.8.5 Typically 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.8.6 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

11.8.7 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

11.8.8 Weigh dish as soon as it has cooled to room temperature.

11.8.9 Return the samples to the oven for another 15 – 20 minutes, cool in a desiccator and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained (weight loss is less than 0.5 mg.) If more than three cycles are required, write a NCM.

11.8.10 Calculate the results using the formula given in Section 12. Use the final weight achieved for calculating VS.

11.9 Volatile Dissolved Solids

11.9.1 Heat muffle furnace up to temperature (550 °C).

11.9.2 Place evaporating dish containing residue generated by Total Dissolved Solids section in muffle furnace to ignite the residue.

11.9.3 Record the date, time, and oven temperature in LIMS when the samples are initially placed in the oven and again when they are removed from the oven.

11.9.4 Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.9.5 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.
11.9.6 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.
11.9.7 Weigh dish as soon as it has cooled to room temperature.
11.9.8 Return the samples to the oven for another 15 – 20 minutes, cool in a desiccator and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained (weight loss is less than 0.5 mg.) If more than three cycles are required, write a NCM.
11.9.9 Calculate the results using the formula given in Section 12. Use the final weight achieved for calculating VDS.

11.10 Volatile Suspended Solids
11.10.1 Heat muffle furnace up to temperature (550 °C).
11.10.2 Place glass fiber filter disc containing residue generated by Total Suspended Solids section in muffle furnace to ignite the residue.
11.10.3 Record the date, time, and oven temperature in LIMS when the samples are initially placed in the oven and again when they are removed from the oven.
11.10.4 Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.
11.10.5 Let filter cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.
11.10.6 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.
11.10.7 Weigh filter as soon as it has cooled to room temperature.
11.10.8 Return the samples to the oven for another 15 – 20 minutes, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained (weight loss is less than 0.5 mg.) If more than three cycles are required, write a NCM.
11.10.9 Calculate the results using the formula given in Section 12. Use the final weight achieved for calculating VSS.

11.11 Settleable Solds
11.11.1 Place 1L of a well mixed sample in a clean Imhoff cone and let settle for 45 minutes.
11.11.2 Gently agitate the cone.
11.11.3 Allow to settle for another fifteen minutes.
11.11.4 Record the volume of settleable solids as mL/L/hr.
11.11.5 If high in solids and sample settles on sides of the cone, wait 30 minutes and stir with a glass rod and record after one hour.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the ST-QAM.

12.2 Calculate **Total Solids** as follows:

$$\text{Total Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

12.3 Calculate **Total Dissolved Solids** as follows:

$$\text{Total Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

12.4 Calculate **Total Suspended Solids** as follows:

$$\text{Total Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

12.5 Calculate **Volatile Solids** as follows:

$$\text{Volatile Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

12.6 Calculate **Volatile Dissolved Solids** as follows:

$$\text{Volatile Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

12.7 Calculate **Volatile Suspended Solids** as follows:

$$\text{Volatile Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + filter before ignition (mg)
B = weight of residue + filter after ignition (mg)
C = volume of sample (mL)

12.8 If multiple weighing cycles are required, the final sample weight is used for calculating solids content.

12.9 If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.

12.10 Calculate Settleable Solids as $\frac{(\text{mls of solids for 1 hr})}{1\text{L}}$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.

13.2.1.1 Project specific requirements, if more stringent than the lab routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – If the method blank concentration exceeds the applicable criteria the samples must be re-prepped/re-analyzed unless their concentration is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in the LIMS.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM, (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.3.2.3 RPD excursion for LCS/LCSD – If spike recoveries are acceptable, data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes.

13.4 Sample Duplicate (DU):

13.4.1 All analytes should be within established control limits for precision (RPD). Limits can be found in LIMS.

13.4.1.1 % RPD excursion for DU - sample/sample duplicate – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM describing the physical evidence; otherwise the sample should be re-prepped and re-analyzed

13.5 Sample Result Evaluation:

13.5.1 Dilutions

13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.5.1.1.1 An NCM is written to document the reason for the dilution.

13.5.1.1.2 If the dilution causes the spike(s) to be diluted out an NCM is written to document the excursion.

13.6 Insufficient Sample:

13.6.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a NCM stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Methods 160.1, 160.2, 160.3, 160.4 and 160.5

17.2 Standard Methods for the Examination of Water and Wastewater, 2011, Methods 2540B, 2540C, 2540D, 2540E, 2540F.

17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs; current revisions:

17.5.1 ST-QA-0002, Standards Preparation

17.5.2 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes

17.5.3 ST-PM-0002, Sample Receipt and Chain of Custody
17.5.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the use of Control Charts
17.5.5 ST-QA-0036, Non-conformance Memorandum (NCM) Process, current revision.
17.5.6 ST-QA-0038, Procedure for Compositing and Subsampling
17.5.7 ST-IP-0004, Glassware Preparation for Inorganic and Trace Metals Analysis

18.0 CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

18.1 Method 2540 B, 2540 C, 2540 D, 2540 E
18.1.1 Samples are not taken through the drying, cooling, and weighing cycle more than three times. If the sample fails to reach constant weight within three cycles, this is documented in a Nonconformance Memo and the final weight is used for the calculation.

18.2 Method 160.2
18.2.1 The filter cleaning procedure outlined in the method is not utilized. This SOP requires the use of method blanks to document system cleanliness.

18.3 Method 160.3, 2540 B, 2540 C, 2540 D, 2540 E
18.3.1 Constant Weight is obtained when weight change is less than 0.5 mg. The 4% option is not utilized.

18.4 Method 2540 B, 2540 C, 2540 D, 2540 E
18.4.1 Lab applies an RPD criteria of 20% for duplicates, not the method criteria that duplicate determinations should agree within five percent of their average.

18.4.1 Work done for the State of South Carolina must meet the method requirement for a 5% RPD between duplicate determinations.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 South Carolina RPD requirement of 5% added in Section 18.4.1
19.2 Rev 11:
19.2.1 No Changes, Annual Review.
19.3 Rev 12: (08/08/2013)
19.3.1 Grammatical corrections throughout
19.3.2 Removal of references to QuantIMS and Clouseau
19.3.3 Section 6, removal of glass beakers for TS or TDS determination
19.3.4 Replaced reagent water and distilled water with DI water throughout
19.3.5 Replaced recording information on bench sheet with recording in LIMS throughout
19.3.6 Updated equipment in Section 6
19.3.7 Section 9, removal of QuantIMS requirements
19.3.8 Updated Section 10 balance calibration requirements
19.3.9 Section 13 updated with corrective actions
19.3.10 Updated Section 15
19.4 Rev 13: (05/29/2015)
19.4.1 Changed wording throughout so all references to non-conformance are called NCM's
19.4.2 Changed acronym for sample duplicate to DU, to match LIMS
19.4.3 Added applicable methods to section 18

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Appendix B
WASTE MANAGEMENT PLAN

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FINAL

WASTE MANAGEMENT PLAN

BASEWIDE GROUNDWATER MONITORING PROGRAM

For

ALAMEDA POINT, ALAMEDA, CALIFORNIA

August 2015

Prepared for

Naval Facilities Engineering Command Southwest
San Diego, California

Prepared Under

Contract N62473-12-D-2012
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MMEC Group Project 5023-14-6096

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ACRONYMS AND ABBREVIATIONS

Alameda Point	former Naval Air Station Alameda
Amec Foster Wheeler	Amec Foster Wheeler Environment & Infrastructure, Inc.
BGMP	Basewide Groundwater Monitoring Program
CFR	Code of Federal Regulations
DON	Department of the Navy
DOT	Department of Transportation
EBMUD	East Bay Municipal Utility District
IDW	investigation-derived waste
IR	Installation Restoration
MMEC Group	Multimedia Environmental Compliance Group
MSW	municipal solid waste
NAVFAC SW	Naval Facilities Engineering Command Southwest
POTW	publicly owned treatment works
PPE	personal protective equipment
RASO	Radiological Affairs Support Office
RCRA	Resource Conservation and Recovery Act
ROICC	Resident Officer in Charge of Construction
RPM	Remedial Project Manager
TCLP	toxicity characteristic leaching procedure
TSDF	treatment, storage, and disposal facility
U.S. EPA	United States Environmental Protection Agency
VOC	volatile organic compound
WMP	Waste Management Plan

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1 WASTE MANAGEMENT SUMMARY

The Multimedia Environmental Compliance Group (MMEC Group), a joint venture comprising Amec Foster Wheeler, Inc. (Amec Foster Wheeler) and KMEA, is supporting the Naval Facilities Engineering Command Southwest (NAVFAC SW) by conducting a Basewide Groundwater Monitoring Program (BGMP) at former Naval Air Station Alameda (Alameda Point), in Alameda, California. This work is being performed under NVAFAC SW Contract Number N62473-12-D-2012, Task Order Number 0096.

This Waste Management Plan (WMP) describes the manner in which investigation-derived waste (IDW) will be handled. The United States Environmental Protection Agency (U.S. EPA) *Management of Investigation-Derived Wastes During Site Inspections,* developed in 1991, will be used as guidance for waste management methods during this project. All subsurface soil, purged groundwater, and decontamination fluids generated as part of the BGMP will be containerized and stored onsite in a secure and controlled area until the material is properly classified for disposal. IDW generated at Installation Restoration (IR) Sites 1, 2, and 32 will be segregated and stored separately from the IDW generated from all other sites because of the possibility of the material containing radionuclides. IDW storage containers will be confirmed to be compatible with the IDW to be generated and stored, and all personnel will wear the appropriate level of personal protective equipment (PPE) while handling wastes. Also, IDW will be stored in an area where damage to the containers is unlikely (such as away from vehicle and equipment high-traffic areas).

IDW includes soil cuttings and other solid waste from well installation or decommissioning, groundwater, decontamination fluids, PPE, and decontamination equipment, which will be managed as follows:

Soil cuttings and solid waste:

- Soil and other solid waste generated during well installation or decommissioning drilling will be stored in Department of Transportation (DOT)-approved 55-gallon drums. A forklift may be used to transport 55-gallon drums filled at the well location to the designated IDW storage area. Following completion of the investigation activities, the soil and solid waste will be characterized and shipped offsite to an appropriate disposal facility. IDW generated from IR Sites 1, 2, and 32 will be segregated and stored separately from IDW generated from the other IR sites being sampled. This waste will be treated as radiological waste, and will be stored within a designated radiological waste storage area until it has been cleared by the Radiological Affairs Support Office (RASO) for disposal. The IDW will then be physically screened out of the area by a radiological technician prior to transport and disposal. A third-party contractor will be used to dispose of all radiological waste generated from the site activities.

Groundwater:

- Purged groundwater and decontamination fluids will be stored temporarily in 5-gallon buckets during sampling. The water will then be transferred to 55-gallon drums or a temporary storage container (polyethylene tank, etc.) at the designated IDW storage area or the designated radiological waste storage area (IR Sites 1, 2, and 32), and drums (or temporary storage container) will be stored within a secondary containment berm. Following completion of the site activities, analytical results from the groundwater sampling will be used to characterize and profile the waste for offsite disposal at an appropriate accepting facility. Groundwater and decontamination fluid generated from IR Sites 1, 2, and 32 will be treated as radiological waste and will be handled as described above.

PPE, decontamination equipment, and miscellaneous non-hazardous solid waste (refuse):

- PPE and sampling equipment IDW will be generated during the field work. Used PPE may consist of protective coveralls, nitrile gloves, and other disposable gear associated with field activities. Sampling equipment may include such items as sampling tools, cleaning/decontamination equipment, and used paper towels. Used PPE will be stored onsite, double-bagged, and disposed of along with other non-hazardous solid waste pending the analytical results of samples or thrown away in the general trash if there is no concern for contamination. PPE generated from IR Sites 1, 2, and 32 will be treated as radiological waste and will be handled as described above.

A solid waste is a Resource Conservation and Recovery Act (RCRA) characteristic hazardous waste if it exhibits the characteristics of ignitability, corrosivity, reactivity, or toxicity, as defined in Title 40 of the Code of Federal Regulations (CFR), Section 261, Subpart C. Toxicity will be determined in accordance with the toxicity characteristic leaching procedure (TCLP). IDW will also be evaluated as a State of California hazardous waste per Title 22.

Soil cuttings and fluids pending characterization will be placed 55-gallon DOT-approved drums. Each drum stored at the site will have a self-adhesive label affixed to the outside of the container. At a minimum, the label will document the following:

- Waste type (e.g., soil, purge water, decontamination fluids)
- Site name—IR Site Numbers
- Well from which the IDW was generated
- Date of accumulation
- Estimated quantity in container (e.g., 50 gallons)
- Navy Resident Officer in Charge of Construction (ROICC) name and telephone number as an emergency contact

Drums containing IDW are to be stored in rows with labels facing outward for easy identification.

The disposal of IDW will be in compliance with the Offsite Rule (40 CFR 300.440) and other pertinent codes and regulations. Hazardous substances will be characterized in accordance with applicable U.S. EPA and state regulations. The MMEC Group will use sample results from the generating groundwater sampling activities to characterize and profile the IDW for transportation and disposal offsite in accordance with the requirements of the disposal facility, and will provide the Navy complete waste manifests and similar documentation. For IR Sites 1, 2, and 32, the MMEC Group will provide the RASO will all analytical results from the groundwater sampling for review and approval for disposal. Once approval has been granted, the IDW will then be physically screened out of the area by a radiological technician prior to transport and disposal. A third-party contractor will be hired to dispose of all radiological waste generated from the site activities.

Disposal of IDW will be performed within 90 days of waste generation. The removal of IDW to an offsite disposal facility will be coordinated with the Navy Remedial Project Manager (RPM), RASO, and site ROICC.

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2 WASTE MANAGEMENT AND DECONTAMINATION AREAS

A temporary IDW staging area will be established onsite to store project waste streams prior to disposal. The area will be the primary area for receiving and temporarily holding project wastes prior to characterization and offsite disposal. Further segregation within these areas will be conducted on the basis of anticipated waste characterization results (i.e., RCRA hazardous versus non-RCRA hazardous versus non-hazardous designation). Note that onsite storage of RCRA and non-RCRA hazardous waste will be limited to fewer than 90 days without a permit.

An existing Alameda Point radiological waste staging area will be also be used for waste originating from IR Sites 1, 2, and 32. All solid and liquid waste generated from these sites will be collected in 55-gallon drums and stored in this secure area pending characterization and disposal approval from RASO.

2.1 SOIL AND OTHER SOLID WASTE STAGING

Soil and other solid materials generated from the proposed groundwater well installation or decommissioning procedures will be collected, properly containerized, and transported to the appropriate staging area. Waste solids from all sites except IR Sites 1, 2, and 32 may be consolidated with other waste solids in 55-gallon drums at the staging area until analytical results have been received and characterization is complete. Waste solids from IR Sites 1, 2, and 32 may be consolidated in 55-gallon drums and stored in the radiological waste staging area.

2.2 WASTEWATER STAGING

Waste groundwater development, purge, and sampling water will be collected, properly containerized, and transported to the appropriate staging area. Waste liquids from all sites except IR Sites 1, 2, and 32 may be consolidated with other waste liquids in the polyethylene tank or in 55-gallon drums at the staging area until analytical results have been received and characterization is complete. Waste liquid from IR Sites 1, 2, and 32 may be consolidated and stored in the radiological waste staging area.

2.3 DECONTAMINATION

Reusable, non-dedicated equipment used for development or sampling (e.g., water-level meter, submersible pump or surge block, and hand-sampling tools) will be decontaminated prior to initial use and after each use by washing with a non-phosphate detergent (e.g., Liquinox™ or equivalent) and rinsed with fresh potable water, followed by rinsing with deionized water. The rinsed equipment will be placed on clean plastic sheeting and allowed to air dry. If equipment will not be reused immediately, it will be placed in plastic bags or covered in clean plastic sheeting until used. Tools and items for which decontamination is difficult or impossible to verify will remain onsite until completion of the work for subsequent packing and offsite disposal at an approved disposal facility.

Residual solids or liquids will be collected, properly containerized (i.e., in DOT-approved 55-gallon drums) and transported to and staged at the appropriate staging area pending characterization and disposal. Waste liquids may be consolidated with other project waste liquids in the existing 55-gallon drums or aboveground wastewater storage tanks at the central storage area, and residual decontamination solids may be consolidated with other solids; however, wastes from IR Sites 1, 2, and 32 must be segregated and consolidated separately in the approved radiological waste staging area.

During well construction or decommissioning, the use heavy equipment will be required, including drill rigs and support vehicles. Heavy equipment and vehicles that come into contact with suspected contaminated soil and/or liquids will be decontaminated prior to demobilization from any individual work site to prevent contaminants from being transferred out of the active work zone. Dry brushing or wiping will be the primary decontamination methods used for equipment and vehicles to minimize the volume of decontamination rinsate water requiring treatment and/or disposal. If necessary, because of heavy contamination or wet conditions, equipment and vehicles (especially tires) will be washed with a pressure washer. Decontamination will proceed until the soil and residues are removed. Decontamination water from these activities will be collected and included in the 55-gallon drums or polyethylene tank designated for that area.

3 WASTE CHARACTERIZATION

Project waste materials will consist of solid and liquid waste and will be stored in appropriate containers (e.g., DOT-approved 55-gallon drums or polyethylene tanks) pending waste characterization and disposal. Wastes that are temporarily stored at the IDW storage area or at the radiological waste storage area will be labeled as “Classification Pending Analysis.”

Characterization of project wastes will be necessary for proper waste profiling and waste acceptance by receiving facilities. To properly characterize wastes, the following sampling conventions will be followed:

- For drummed solid waste (e.g., soil), a well-mixed, composite sample will be collected from several areas within the drum (one composite sample per drum) using a stainless-steel sampling trowel, a stainless-steel hand auger or coring device, or equivalent.
- For containerized wastewater, analytical results from the groundwater sampling event will be used to characterize the storage containers or a composite wastewater sample will be collected and analyzed.

Waste characterization samples will be collected and placed in laboratory-provided glassware and shipped to the designated California-certified and Department of Navy (DON)-approved offsite laboratory. Analytical criteria for waste characterization samples will depend on the requirements of the receiving facility. For example, the analytical criteria considered likely for proper profiling of soil waste will include:

- Metals (TCLP if necessary)
- Volatile organic compounds (VOCs)

Specific sampling requirements for each well can be found in the Sampling and Analysis Plan.

Several options for discharge of wastewater include discharge to the publicly owned treatment works (POTW) via sanitary sewer or discharges to the storm sewer or offsite treatment and discharge facility. In the case of using the sanitary sewer, the discharge must meet the wastewater discharge standards established by East Bay Municipal Utility District (EBMUD) Ordinance No. 311A-03. An appropriate suite of analytes will likely be analyzed for wastewater samples to meet storm water discharge or offsite treatment, storage, and disposal facility (TSDF) requirements. Additional tests may also be required on the basis of the results of the tests above and discussions with the receiving facilities. Note that prior Alameda Point projects used either the local POTW or offsite TSDFs for disposal of wastewater from site activities. All wastewater generated from IR Sites 1, 2, and 32 will be transported to an offsite regulated facility and will not be discharged to the local POTW.

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4 WASTE PROFILING AND DISPOSAL

Based on the results of the waste characterization analyses, project wastes will be properly profiled for disposal at an appropriate California-licensed disposal facility.

General waste, including used PPE, disposable sampling tools, paper towels, and other incidentals, that is not visibly contaminated or grossly soiled will be disposed of as municipal solid waste (MSW) in an appropriate MSW receptacle at each project work site. These receptacles will be emptied and their contents disposed of through existing MSW pick-ups for the particular work site. Alternatively, these general wastes will be consolidated in an appropriate MSW receptacle at an individual project work site with an active contract for MSW hauling.

For project wastewater, if the results of the waste characterization analyses meet surface-water discharge standards, the water may be discharged to the site sewer system. Project wastewater that does not meet surface-water discharge standards but does meet POTW acceptance criteria may be discharged to the POTW through the sanitary sewer system. Project wastewater that does not meet surface-water or POTW discharge standards will be transported to an offsite treatment and disposal facility on the basis of characterization data and the associated waste profile. It is assumed that non-hazardous project wastewater requiring offsite disposal will be transported to and disposed of at the Oakland EBMUD treatment facility under the Alameda Point Basewide Program's Trucked Non-Hazardous Waste Permit # INTE3000-001.

Solid wastes (soil, debris, and grossly soiled or visibly contaminated used PPE or sampling tools) generated during the BGMP will be transported to and disposed of at an appropriate offsite landfill facility on the basis of characterization data and the associated waste profile.

All solid and liquid waste generated from IR Sites 1, 2, and 32 will be classified in accordance with 49 CFR. Results will be provided to RASO for review and approval for disposal. Once approval has been granted, the IDW storage containers will be physically screened out of the area by a radiological technician prior to transport and disposal. A third-party contractor will be used to dispose of all radiological waste generated from the site activities.

The waste hauling subcontractor will supply the labor, equipment, and materials necessary to transport the wastes. Individual drivers must be employees of the selected subcontractor.

The MMEC Group will verify or alert the identified waste disposal facilities. The ultimate receiving facility will issue a letter of waste acceptance for each waste stream, as indicated by waste characterization information and the associated profile.

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Appendix C
ENVIRONMENTAL PROTECTION PLAN

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FINAL

ENVIRONMENTAL PROTECTION PLAN

BASEWIDE GROUNDWATER MONITORING PROGRAM

For

ALAMEDA POINT, ALAMEDA, CALIFORNIA

August 2015

Prepared for

Naval Facilities Engineering Command Southwest
San Diego, California

Prepared Under

Contract N62473-12-D-2012
Task Order 0096

DCN: MMEC-2012-0096-0003

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MMEC Group Project 5023-14-6096

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ACRONYMS AND ABBREVIATIONS

Amec Foster Wheeler	Amec Foster Wheeler Environment & Infrastructure, Inc.
BGMP	Basewide Groundwater Monitoring Plan
BMP	best management practice
CASQA	California Stormwater Quality Association
CDFW	California Department of Fish and Wildlife
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CNDDB	California Natural Diversity Database
EPP	Environmental Protection Plan
FAR	Federal Acquisitions Regulation
FP	Fully Protected
IDW	investigation-derived waste
IR	Installation Restoration
ITSI	Innovative Technical Solutions, Inc.
MGP	manufactured gas plant
MMEC Group	Multimedia Environmental Compliance Group
NAS	Naval Air Station
NAVFAC SW	Naval Facilities Engineering Command Southwest
Navy	Department of the Navy
OU	Operable Unit
PCB	polychlorinated biphenyl
PM	Project Manager
PPE	personal protective equipment
RASO	Radiological Affairs Support Office
RPM	Remedial Project Manager
SSC	Species of Concern
SWRCB	State Water Resources Control Board (California)
USFWS	United States Fish and Wildlife Service
Water Board	California Regional Water Quality Control Board, San Francisco Bay Region
WL	Watch List

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1 INTRODUCTION

1.1 GENERAL OVERVIEW AND PURPOSE

This Environmental Protection Plan (EPP) has been prepared for conducting a Basewide Groundwater Monitoring Program (BGMP) at former Naval Air Station Alameda (Alameda Point), in Alameda, California (Figure C-1). This EPP has been prepared by the Multimedia Environmental Compliance Group (MMEC Group), a joint venture between Amec Foster Wheeler, Inc. (Amec Foster Wheeler) and KMEA, and is being performed under Naval Facilities Engineering Command Southwest (NAVFAC SW) Contract Number N62473-12-D-2012, Task Order Number 0096.

This EPP identifies known or potential environmental issues that may be encountered while conducting the BGMP and presents mitigation measures and best management practices (BMPs) that will be implemented.

1.2 GENERAL SITE INFORMATION

Alameda Point is on the western tip of Alameda Island, which is on the eastern side of San Francisco Bay (Figure C-1). Most of the northern portion of what is now Alameda Point was covered by the waters and tidal lands of San Francisco Bay. To create Alameda Point, fill material was dredged from San Francisco Bay. In 1930, the U.S. Army acquired Alameda Point from the City of Alameda. In 1936, the Department of the Navy (Navy) acquired the land from the U.S. Army and built the former NAS Alameda, which was operated as an active naval aircraft maintenance, repair, and refit facility from 1940 to 1997. During the history of the former Naval Air Station (NAS) Alameda, it housed approximately 60 military tenant commands for a combined military and civilian work force of over 18,000 personnel. Upon closure in April 1997, the site was renamed Alameda Point in a reorientation of the facility toward civilian use. In July 1999, Alameda Point was identified as a National Priorities List site and the Navy is currently conducting a site investigation in accordance with Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

From the late 1800s until the 1920s, two manufactured gas plants and an oil refinery located near present-day Alameda Point apparently discharged petroleum products and other wastes to adjacent marshlands. These wastes then spread through tidal action over the marsh surface and tidal flats. As the marshlands and subtidal areas were filled, the discharged petroleum products became entrapped in the subsurface, creating what is now referred to as the Marsh Crust and subtidal area (Bechtel, 2008). The Marsh Crust and subtidal area material consists of a thin (up to 6-inch thick) layer of hydrocarbon-contaminated sediment indicative of coal-based manufactured gas plant (MGP)-derived waste.

Wastes historically disposed of at the site included solvents, waste oil, paints, scrap metal, garbage, radiological material, aircraft engines, and possibly munitions and explosives of concern. In addition, it is estimated that 365,000 gallons of aviation fuel have leaked from underground fuel tanks (Innovative Technical Solutions, Inc. [ITSI], 2008). Numerous plating and painting shops are also being investigated to determine the extent of contamination resulting from previous spills and other disposal practices.

Various Installation Restoration (IR) sites have been defined at Alameda Point, some of which are subject to BGMP activities. In some cases, multiple IR sites were grouped into various Operable Units (OUs) (Figure C-2). The Alameda Point BGMP provides groundwater and soil gas analytical data to be used, in conjunction with other information generated by ongoing site investigation and remedial actions, to define the nature and extent of contamination in groundwater at Alameda Point (ITSI, 2008). A number of individual or commingled groundwater plumes have been identified at Alameda Point. The groundwater plumes generally consist of chlorinated constituents, petroleum hydrocarbons and other fuel-related compounds, metals, or a combination of these contaminants (ITSI, 2008). To monitor these plumes, groundwater samples are routinely collected from selected groundwater monitoring wells and are analyzed for site-specific analytes.

2 ENVIRONMENTAL RESOURCE PROTECTION AND MITIGATION

This section identifies the protection measures that will be implemented to avoid, minimize, and mitigate potential impacts from the BGMP activities to special-status wildlife.

2.1 THREATENED, ENDANGERED, AND SENSITIVE SPECIES

The California Natural Diversity Database (CNDDB) was queried for known occurrences of federal- or state-listed threatened and endangered species (California Department of Fish and Wildlife ([CDFW], 2015a and 2015b). The CNDDB identified the California least tern (*Sterna antillarum browni*) as occurring at Alameda Point, as well as several other special-status species that may be encountered during BGMP activities. The results of the query are shown in Table 2-1.

For the purposes of this EPP, only avian special species will be addressed. Species such as the California tiger salamander (*Ambystoma californiense)* and the Salt-marsh harvest mouse (*Reithrodontomys raviventris*) are unlikely to be encountered in the specified work areas during BGMP activities. No work will be performed within the waters surrounding Alameda Point; therefore, sensitive fisheries will be avoided. No vegetation will be disturbed during existing monitoring well sampling activities. If proposed drilling activities will be performed in a non-disturbed area with the potential to contain any of the three species of plants listed in Table 2-1, i.e., Santa Cruz tarplant, Beach layia, and Robust spineflower, then the possibility for and necessity of a focused biological survey will be discussed with the NAVFAC SW Remedial Project Manager (RPM).

Table 2-1
Results of California Natural Diversity Database Query for Alameda Point Vicinity

Common Name	Scientific Name	Federal Status	State Status	CDFW Status
Amphibians				
California tiger salamander	*Ambystoma californiense*	Threatened	Threatened	SSC
Birds				
Cooper's hawk*	*Accipiter cooperii*	None	None	WL
Northern harrier*	*Circus cyaneus*	None	None	SSC
White-tailed kite*	*Elanus leucurus*	None	None	FP
Alameda song sparrow*	*Melospiza melodia pusillula*	None	None	SSC
California least tern[2]*	*Sternula antillarum browni*	Endangered	Endangered	FP
Saltmarsh common yellowthroat*	*Geothlypis trichas sinuosa*	None	None	SSC
California brown pelican*	*Pelecanus occidentalis californicus*	Delisted	Delisted	FP
Double-crested cormorant*	*Phalacrocorax auritus*	None	None	WL
California black rail[2]*	*Laterallus jamaicensis coturniculus*	None	Threatened	FP
California clapper rail[2]*	*Rallus longirostris obsoletus*	Endangered	Endangered	FP
Burrowing owl*	*Athene cunicularia*	None	None	SSC
American peregrine falcon[1]*	*Falco peregrinus anatum*	Delisted	Delisted	FP
Canada goose[1]*	*Branta canadensis*	None	None	–
Fish				
Tidewater goby	*Eucyclogobius newberryi*	Endangered	None	SSC
Mammals				
Salt-marsh harvest mouse	*Reithrodontomys raviventris*	Endangered	Endangered	FP
Alameda Island mole	*Scapanus latimanus parvus*	None	None	SSC
Pallid bat	*Antrozous pallidus*	None	None	SSC
Plants				
Santa Cruz tarplant	*Holocarpha macradenia*	Threatened	Endangered	-
Beach layia	*Layia carnosa*	Endangered	Endangered	-
Robust spineflower	*Chorizanthe robusta var. robusta*	Endangered	None	-

Notes:

1. Species not listed in CNDDB; however, previous site visits have indicated the presence of this species within the Alameda Point area.
2. Species is considered a sensitive species at Alameda Point for this BGMP EPP.

* Species protected by the Migratory Bird Treaty Act of 1918 as of December 2, 2013

CDFW = California Department of Fish and Wildlife

FP = Fully Protected

SSC = Species of Special Concern

WL = Watch List

2.1.1 California Least Tern

The California least tern is listed by the California Department of Fish and Wildlife (CDFW) as a fully protected species and by the United States Fish and Wildlife Service (USFWS) as an endangered species. The California least tern is also protected under the Migratory Bird Treaty Act (16 United States Code 703-711). California least terns are migratory and typically arrive in California in April and May. They feed diurnally, primarily on small marine fish species. Breeding colonies are generally located in abandoned salt ponds and along estuarine shorelines that are relatively free of predators and human disturbance. Nests are arranged in loose colonies on barren to sparsely vegetated substrate (usually sand and gravel) near the shoreline. Nesting colonies typically separate in late July to mid-August.

A large colony of California least tern have been documented nesting in the former airfield at Alameda Point (Figure C-3). A minimum non-disturbance buffer zone of approximately 1,300 feet (one-quarter mile) will be upheld around this area during the length of the nesting season (April 1 – August 31).

2.1.2 Other Species

In addition to the California least tern, several other species, including the California clapper rail (*Rallus longirostris obsoletus)* and the California black rail (*Laterallus jamaicensis coturniculus*), may also occur at Alameda Point. Both of these species are protected under the federal and/or state endangered species acts and have the potential to be found within emergent salt marsh habitats at Alameda Point (i.e., cordgrass). Work within these habitats will be avoided. If potential habitat for these species occurs near drilling activities, a 500-foot non-disturbance area will be upheld for the potential habitat during the nesting season (February 1 – August 31). In addition, all avian species present at Alameda Point are currently protected under the Migratory Bird Treaty Act as of December 2, 2013. If active nests of other avian species are encountered, they will be avoided with a minimum 100-foot buffer zone.

Planned BGMP sampling, well installation, and well decommissioning planned for Alameda Point are not anticipated to have an adverse effect on the species mentioned above. When feasible, work near potential nesting sites will be conducted outside of the avian nesting season, which is generally defined as February 1 – August 31.

2.2 MITIGATION MEASURES

The MMEC Group will fully implement and adhere to the following protection measures to avoid, minimize, and mitigate potential impacts from the planned BGMP activities.

2.2.1 Training

The MMEC Group will conduct a training session prior to the start of onsite activities for all MMEC Group personnel and subcontractors. The training will include a discussion of the biology and ecology of the site, threats, and the protection measures provided in this EPP. All MMEC Group personnel and subcontractors working onsite will be required to participate in the orientation training and sign a form stating that the training was attended and that the worker understands all protection measures in place.

The following guidelines will be given:

- Instruction will be given that no personnel working on the BGMP will take or destroy wildlife, including birds, eggs, or active nests.

- Prior to starting work at each well location, active avian nests (i.e., with visible eggs/chicks), will be documented and an appropriate non-disturbance buffer zone will be established around each that will be maintained during the course of all activities at that site location, or until the nests have reached their natural end.
- Any person who inadvertently kills or injures wildlife will be required to immediately report the incident to the MMEC Group Site Supervisor and Project Manager (PM). The MMEC Group PM will then notify the NAVFAC SW RPM, who will then be required to notify the USFWS and/or the CDFG.
- The RPM will be notified verbally and in writing within 24 hours of the accidental death or injury to any state- or federally listed species during the BGMP-related activities. Notification will include summary information about the incident, the type of animal, and any other applicable information. The notification will be forwarded to the USFWS and/or CDFG.
- Avoidance measures will be implemented to minimize and avoid impacts to sensitive habitats and species at Alameda Point. BGMP-related activities will remain within the designated work areas and access roads. Approved work areas, access roads, and staging areas will be clearly marked. No MMEC Group personnel or subcontractors may access or disturb lands outside of the designated work areas.
- Littering is prohibited and personnel will be instructed to dispose of trash in designated containers. All trash and debris will be removed at the end of each work day.
- All MMEC Group personnel and subcontractors will practice fire prevention and fire safety through proper equipment maintenance and adherence to specific site safety procedures.
- Any and all feeding of wildlife by MMEC Group personnel and subcontractors will be prohibited.
- All site personnel must understand the applicable environmental requirements prior to the start of all BGMP activities. It is the responsibility of each person working on the project to understand and comply with the environmental requirements associated with the project. All questions are to be directed to the MMEC Group PM or Site Supervisor.
- All training records will be documented and maintained with the onsite project files.

2.2.2 Work Area Delineation

The area of disturbance for all sampling and drilling related to well installation and decommissioning will be confined to the smallest area possible, and previously disturbed areas will be used as much as practicable. All onsite personnel will access each work area using existing routes and will not cross special-status species' habitat outside of the work area. Previously disturbed and designated areas within the boundaries of the BGMP Alameda Point work area will be used for temporary storage areas and staging areas. Vehicle speeds will not exceed 20 miles per hour to avoid disturbing or injuring special-status species and their habitat while in the work area. If travel off of existing routes will be required, it will be coordinated with and approved in advance by the NAVFAC SW RMP.

2.2.3 Work Area Inspection and Monitoring

Prior to the start of any sampling or drilling activities at a specific work area, the MMEC Group Site Supervisor will visually inspect the area to ensure that there is no presence of special-status species or an active avian nest in or near the selected work area. If nesting birds are discovered, all work activities will be reevaluated to include a non-disturbance buffer zone around each nest that will be maintained during the course of all activities at that site location, or until the nests have reached their natural end. In addition, all drilling locations will be selected to minimize habitat disturbance.

2.2.4 Hazardous Materials and Refuse

Any fuel or hazardous waste leaks or spills that occur onsite will be stopped and repaired immediately and cleaned up at the time of the release. All investigation-derived waste (IDW) will be properly containerized, sealed, and stored in a designated staging area until characterization and disposal. All IDW generated from IR Sites, 1, 2, and 32 will be properly containerized, sealed, and stored in a designated radiological waste storage area until characterization and disposal. No IDW will remain onsite for a period longer than 90 days.

All trash and food items will be secured in a closed container, and all trash and debris will be removed from the work areas after all work has been completed at that locations, or at the end of the work day.

Further descriptions of all waste management activities can be found in Appendix B of the Work Plan (Waste Management Plan).

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3 PREVENTION OF RELEASES TO THE ENVIRONMENT

This section summarizes procedures to be followed to prevent the release of contaminants to the environment during work activities.

Fueling and lubrication of equipment and motor vehicles will be conducted in a manner that protects against spills and evaporation. All fueling and equipment maintenance will take place within existing paved areas, and not closer than 100 feet to any sensitive species, wetlands, or drainage areas. BMP NS-9, Vehicle and Equipment Fueling, will be followed for these practices (California Stormwater Quality Association [CASQA], 2009). Fuels, lubricants, and chemical wastes to be discarded will be managed in accordance with federal, state, and local regulations, including Title 40 of the *Code of Federal Regulations* (CFR), parts 263, 264, 265, 268, and 279. All used oil will be recycled or properly disposed of.

Site work will proceed with specified precautions to prevent releases/spills of oil and hazardous substances. BMP WM-4, Spill Prevention and Control, will be followed as required (CASQA, 2009). All equipment used during the BGMP activities will be inspected for leaks prior to the start of any operation and will be immediately repaired if a leak is discovered. Fueling and maintenance of vehicles and equipment will be performed in accordance with BMP NS-9, Vehicle and Equipment Fueling, and BMP NS-10, Vehicle and Equipment Maintenance (CASQA, 2009).

3.1 NOTIFICATIONS IN THE EVENT OF A RELEASE TO THE ENVIRONMENT

In the event of a release of oil, hazardous substance, chemical, or gas, the Project Manager will immediately notify the Base Fire Department, the NAVFAC SW Contracting Officer's Representative, and the NAVFAC SW RPM. Spill response will be in accordance with 40 CFR 300 and applicable state and local regulations. The MMEC Group will be responsible for verbal and written notifications as required by 40 CFR 355, state and local regulations, and NAVFAC SW instructions. Copies of the written notification and documentation that a verbal notification was made will be provided to the NAVFAC SW within 20 days.

3.2 PROTOCOL FOR UNFORESEEN HAZARDOUS MATERIAL

If materials containing polychlorinated biphenyls (PCBs), lead paint, friable or non-friable asbestos (not previously identified), or any other hazardous material or waste are encountered during fieldwork, then that portion of the work will be stopped and the NAVFAC SW Contracting Officer's Representative will be notified immediately.

The NAVFAC SW RPM will determine whether the material is hazardous after review of contractor-provided analytical data. If the material is determined to be non-hazardous or to pose no danger, then the NAVFAC SW Contract Officer's Representative will provide the notice to the NAVFAC SW contractor to proceed as planned. If material is determined to be hazardous and handling of such material is necessary to accomplish the work, NAVFAC SW may choose to issue a modification pursuant to Federal Acquisition Regulation (FAR) 52.243-4, "Changes" and FAR 52.236-2, "Differing Site Conditions," or to leave the material in place.

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4 PROTECTION OF THE ENVIRONMENT FROM WASTE

4.1 CONTROL AND DISPOSAL OF INVESTIGATION-DERIVED WASTE

All subsurface soil, purged groundwater, and decontamination fluids generated as part of the BGMP activities will be containerized and stored onsite in a secure and controlled area until the material is properly classified for disposal. IDW generated at IR Sites 1, 2, and 32 will be segregated and stored separately from the IDW generated from all other sites because of the possibility of the material containing radionuclides. All IDW will be properly labeled and will be disposed of within 90 days.

IDW includes soil cuttings and other solid waste from well installation or decommissioning, groundwater, decontamination fluids, personal protective equipment (PPE), and decontamination equipment, which will be managed as follows:

Soil cuttings and solid waste:

- Soil and other solid waste generated during well installation or decommissioning drilling will be stored in Department of Transportation-approved 55-gallon drums. A forklift may be used to transport 55-gallon drums filled at the well location to the designated IDW storage area. Following completion of the investigation activities, the soil and solid waste will be characterized and shipped offsite to an appropriate disposal facility. IDW generated from IR Sites 1, 2, and 32 will be segregated and stored separately from IDW generated from the other IR sites being sampled. This waste will be treated as radiological waste, and will be stored within a designated radiological waste storage area until it has been cleared by the Radiological Affairs Support Office (RASO) for disposal. The IDW will then be physically screened out of the area by a radiological technician prior to transport and disposal. A third-party contractor will be used to dispose of all radiological waste generated from the site activities.

Groundwater:

- Purged groundwater and decontamination fluids will be stored temporarily in 5-gallon buckets during sampling. The water will then be transferred to 55-gallon drums or a temporary storage container (polyethylene tank) at the designated IDW storage area or the designated radiological waste storage area (IR Sites 1, 2 and 32). Following completion of the site activities, analytical results from the groundwater sampling will be used to characterize and profile the waste for offsite disposal at an appropriate accepting facility. Groundwater and decontamination fluid generated from IR Sites 1, 2, and 32 will be treated as radiological waste and will be handled as described above.

PPE, decontamination equipment, and miscellaneous non-hazardous solid waste (refuse):

- PPE and sampling equipment IDW will be generated during the field work. Used PPE may consist of protective coveralls, nitrile gloves, and other disposable gear associated with field activities. Sampling equipment may include such items as sampling tools, cleaning/decontamination equipment, and used paper towels. Used PPE will be stored onsite, double-bagged, and disposed of along with other non-hazardous solid waste pending the analytical results of samples or in general trash if there is no suspected contamination. PPE generated from IR Sites 1, 2, and 32 will be treated as radiological waste and will be handled as described above.

4.1.1 Storage, Labeling, Transportation, and Disposal of Waste

The MMEC Group will be responsible for collecting, labeling, containerizing, and characterizing all IDW generated from the BGMP activities. All potentially radioactive IDW will be cleared for disposal by RASO and screened offsite by a radiological technician. No hazardous waste will be stored onsite for more than 90 days. Management of hazardous wastes must meet the specifications as cited in the contract and all applicable federal, state, and local laws and regulations. Prior to removal of any hazardous waste from site, all hazardous waste manifests must be signed by authorized NAVFAC SW personnel.

4.1.2 Recycling of Waste Materials

The MMEC Group will provide clearly marked containers and bins to facilitate recycling of waste materials. Contamination of recyclable materials from incompatible products and materials will be prevented. Separation of IDW at the project site will be accomplished by one of the following methods:

- *Source Separated Method*: Waste products and materials that are recyclable will be separated from trash and sorted into appropriately marked, separate containers and then transported to appropriate recycling facilities for further processing. Trash will be transported to a landfill or incinerator.
- *Co-mingled Method*: All IDW waste will be placed into a single container and then transported to a recycling facility, where the recyclable materials will be sorted and processed and the remaining trash transported to a landfill or incinerator.

4.1.2.1 Used Oil

Fueling and lubrication of equipment will be done in a manner that affords maximum protection against spills and evaporation. Fuels, lubricants, and chemical wastes to be discarded (hazardous waste) will be disposed of in accordance with the approved procedures meeting federal, state, and local regulations, especially 40 CFR 263, 40 CFR 264, 40 CFR 265, 40 CFR 268, and 40 CFR 279.

4.1.3 Pollution Prevention/Hazardous Waste Minimization Procedures

The MMEC Group will make every effort to eliminate or reduce acquisition of products containing hazardous substances or toxic chemicals. By efficiently preventing pollution at its sources, the MMEC Group will achieve cost savings, increase operations efficiencies, improve the quality of services, maintain a safe and healthy work place for employees, and improve the environment. The MMEC Group will comply with the Emergency Planning and Community Right-to-Know Act of 1986 emergency planning and response requirements.

The MMEC Group will follow these hazardous waste minimization practices to reduce wastes:

- Control hazardous materials
- Substitute materials/products
- Process modification/change
- Properly identify hazardous waste
- Delist previously listed hazardous waste
- Segregate waste
- Recycle and reuse

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5 POLLUTION CONTROL PLAN

During all BGMP activities, the work area will be delineated with traffic cones, signs, caution tape, or similar items to prevent unauthorized personnel from entering. Each work area will also be visually inspected by the MMEC Group Site Supervisor prior to the start of all activities to identify the environmental and biological conditions within and surrounding the work area. This will ensure that all personnel working onsite are aware of the surrounding conditions and precautions can be implemented for the safety of the workers and the environment. Other control measures that will be implemented at Alameda Point during BGMP activities are discussed in the following sections.

5.1 STORMWATER MANAGEMENT AND EROSION CONTROL

Construction projects with a disturbed area of less than 1 acre are not covered under the State Water Resources Control Board's ([SWRCB] "General Permit for Stormwater Discharges Associated with Construction and Land Disturbance Activities" (SWRCB Order No. 2009-0009-DWQ). Therefore, a Stormwater Pollution Prevention Plan is not required by the California Regional Water Quality Control Board, San Francisco Bay Region (Water Board) for this project.

Although a Stormwater Pollution Prevention Plan will not be submitted with the Work Plan for the BGMP activities, BMP methods for stormwater runoff control will be included in this EPP. A copy of this EPP will be kept at the onsite office and updated, as required, to reflect current site conditions.

BMPs using a combination of erosion and sediment controls that significantly reduce or prevent potential pollutants from runoff from the Alameda Point BGMP will be used during all phases of the project, but specifically during drilling associated with well installation and decommissioning activities. These controls may include erosion control measures, sand bags, vegetation preservation, street sweeping, and material management (CASQA, 2009).

The following BMP methods may be used to prevent erosion and control sedimentation during the BGMP activities:

- Erosion control – scheduling, preservation of existing vegetation
- Sediment control – gravel bagberms, sandbag barrier, and wind erosion control
- Wind erosion – spill prevention and control
- Temporary materials management – solid waste management, hazardous waste management, IDW management, sanitary-septic waste management, and vehicle and equipment fueling and repairs

These BMPs will be implemented at the start of the project and will remain in effect until the designated work has been completed.

5.2 DUST CONTROL

Dust and particulates will be controlled to minimize contaminant spread and to protect human health and the environment. Dust control at Alameda Point during the BGMP implementation will be achieved by keeping dust down at all times, including during non-working periods. Work activities will be designed to control and minimize particulate emissions. Control of particulates will involve cleaning loose dirt from all vehicles used during BGMP activities, enforcing speed limits on roadways, and ensuring that all solid IDW is properly stored and covered.

Although less than 1 acre will be disturbed during the BGMP activities, BMP methods for stormwater runoff will meet the requirements of SWRCB Order No. 2009-0009-DWQ.

5.3 NOISE CONTROL

Field equipment approved for this project will make use of low-noise emission products and equipment, as certified by the United States Environmental Protection Agency.

5.4 MUD AND SEDIMENT CONTROL

Mud and sediment control will be applied during all drilling activities. Precautions will be taken to ensure that all soils generated from drilling activities will be immediately placed into a 55-gallon drum or other container to limit transport of that material. Once drilling activities have been completed, the MMEC Group will visually inspect the area for loose debris or soil. All material discovered will be removed and the area cleaned. This will limit the amount of sediment that may be transported to surrounding areas, storm drains, or watercourses.

5.5 SEVERE WEATHER

The NAVFAC SW RPM will have the authority to stop all fieldwork because of inclement weather and/or natural disasters.

6 REFERENCES

Bechtel, 2008, Final Remedial Investigation Report for Operable Unit 2C, Alameda Point, Alameda, California, September.

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California Stormwater Quality Association (CASQA). 2009. *Stormwater Best Management Practice Handbook, Construction*. September.

Innovative Technical Solutions, Incorporated (ITSI). 2008. Final Annual Groundwater Monitoring Report, Basewide Groundwater Monitoring Program, Alameda Point, Alameda, California. November.

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FIGURES

Figure C-1 Facility Location Map

Figure C-2 Operable Unit and Installation Restoration Site Locations

Figure C-3 California Least Tern Colony Nesting Grounds

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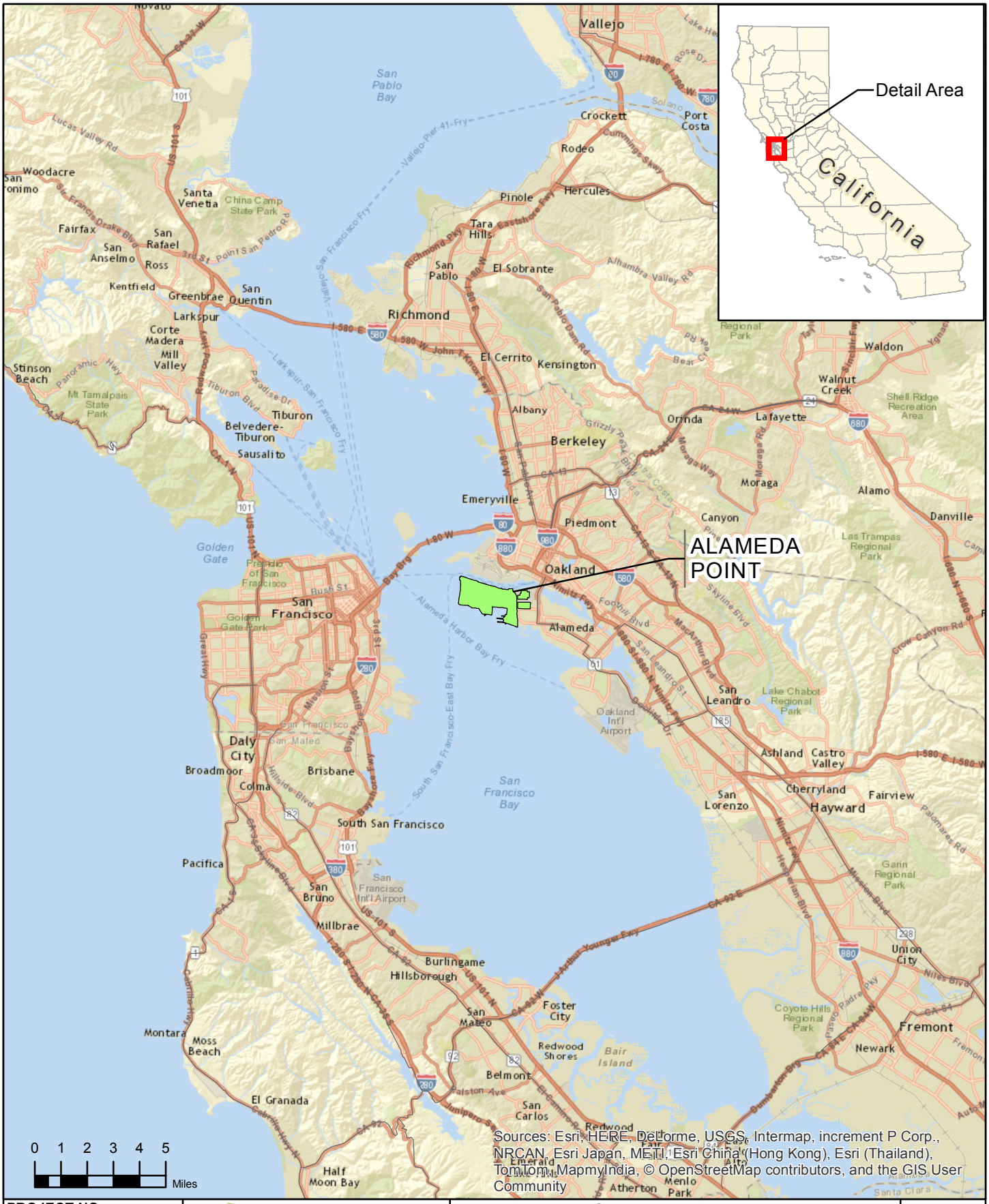

PROJECT NO.: 5023-14-6096	MNEC GROUP	Basewide Groundwater Monitoring Program Alameda Point, Alameda, California	FIGURE
DATE: FEBRUARY 2015			
DRAWN BY: SB		FACILITY LOCATION MAP	C-1
CHECKED BY: KO			

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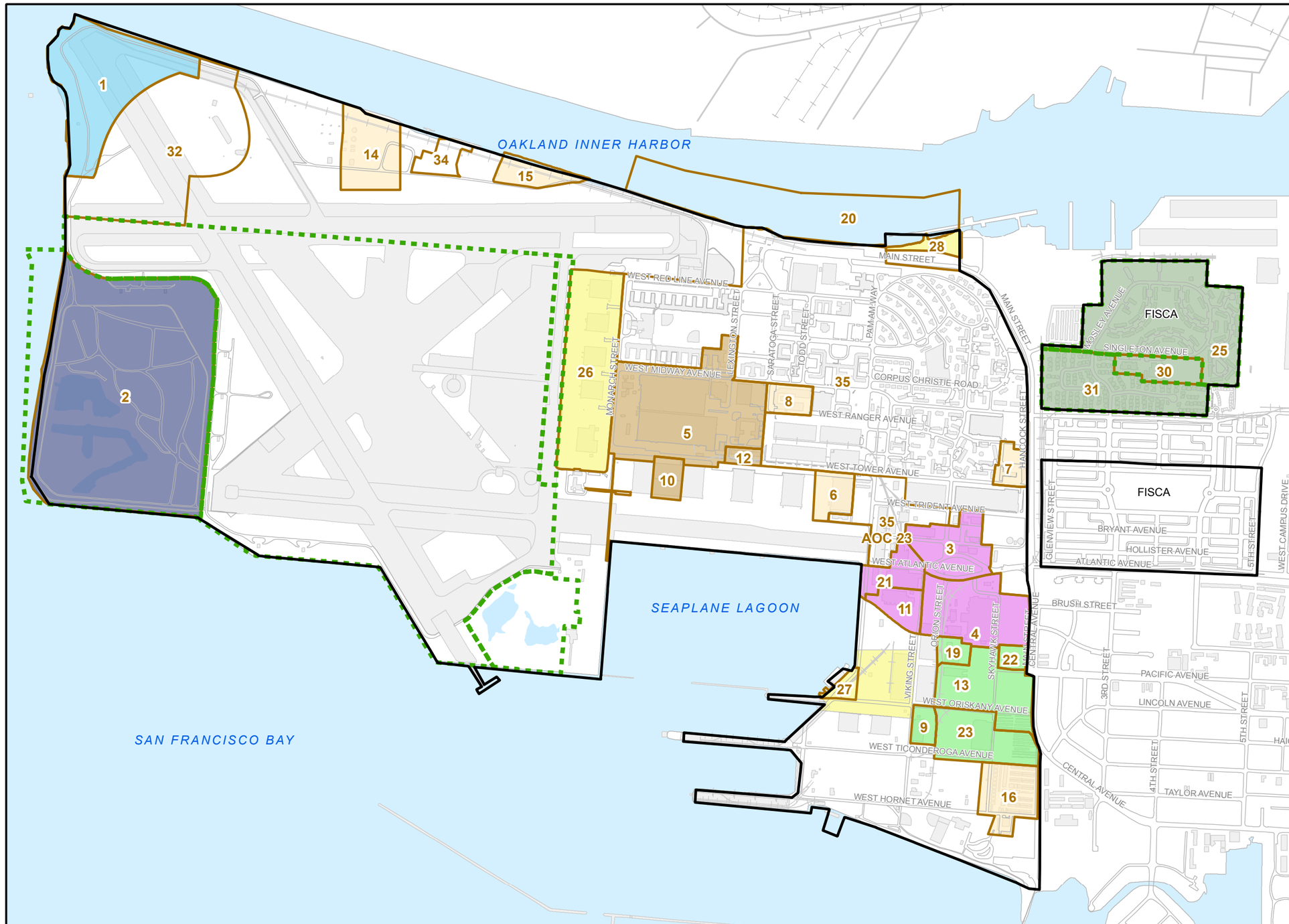

Legend

- Alameda Point Boundary
- Facility Infrastructure
- Railroad
- 1 Installation Restoration Site and Number
- Operable Unit 1
- Operable Unit 2A
- Operable Unit 2B
- Operable Unit 2C
- Operable Unit 3
- Operable Unit 4A
- Operable Unit 5
- Operable Unit 6
- Federal Transfer Parcel

Notes:

FISCA = Fleet and Industrial Supply Center Alameda

0 1,200 2,400 Feet

N

PROJECT NO.:	5023-14-6096
DATE:	FEBRUARY 2015
DRAWN BY:	SB
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MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

OPERABLE UNIT AND INSTALLATION RESTORATION SITE LOCATIONS

FIGURE C-2

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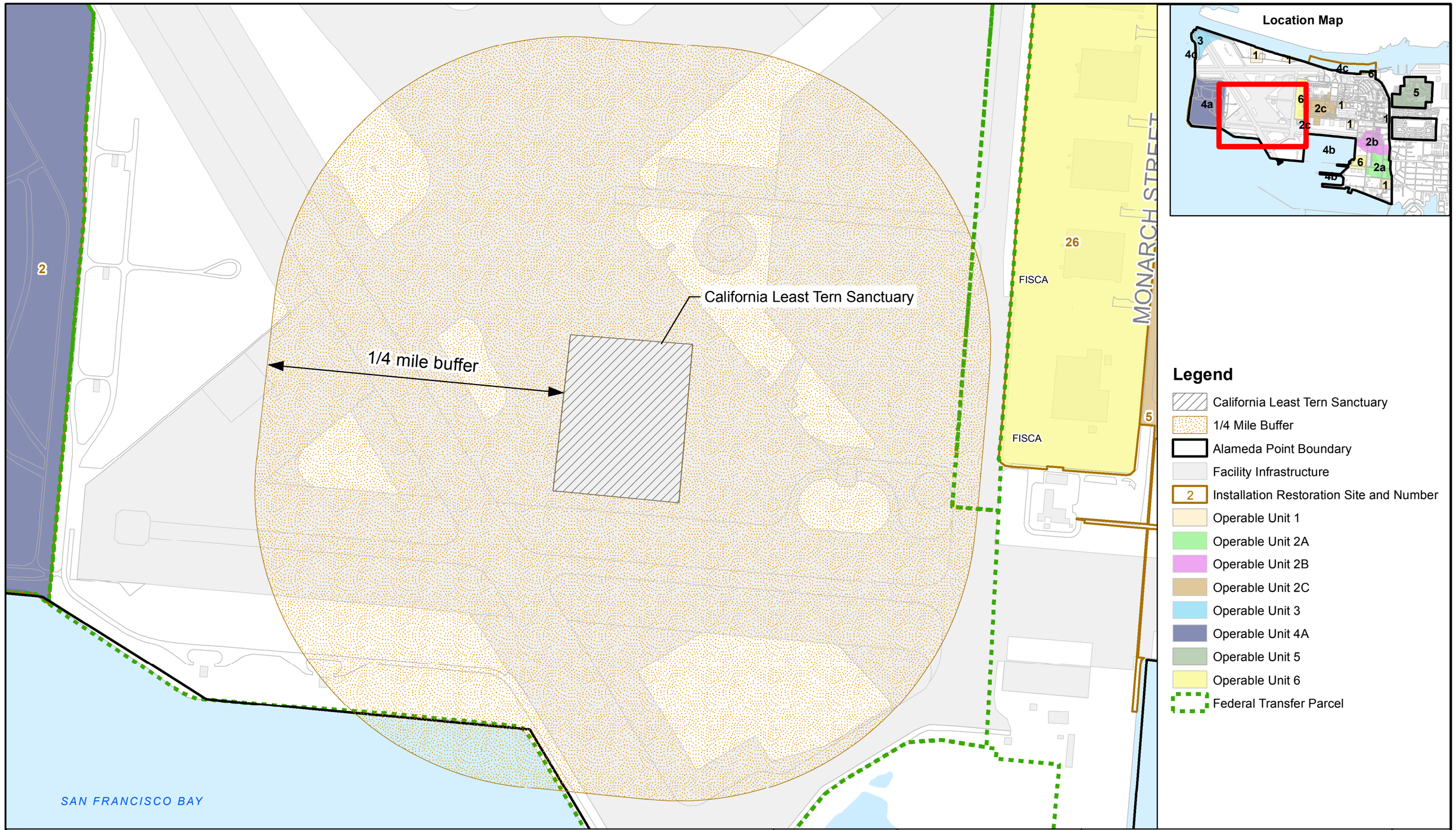

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DATE:	FEBRUARY 2015
DRAWN BY:	SB
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MMEC GROUP

Basewide Groundwater Monitoring Program
Alameda Point, Alameda, California

CALIFORNIA LEAST TERN COLONY NESTING GROUNDS

FIGURE C-3

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