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FINAL

REMEDIAL INVESTIGATION WORK PLAN FOR THE DEFENSE REUTILIZATION AND MARKETING OFFICE SOUTH AND CRANE TEST AREA NORTH

FORMER MARE ISLAND NAVAL SHIPYARD VALLEJO, CALIFORNIA

November 2013

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ARCADIS prepared this plan at the direction of the United States Department of the Navy Base Realignment and Closure (BRAC) Program Management Office (PMO) West. This document should be used only with the approval of BRAC PMO West. This plan is based, in part, on information provided in other documents and is subject to the limitations and qualifications presented in the referenced documents.

November 2013

EXECUTIVE SUMMARY

On behalf of the United States Department of the Navy (Navy), ARCADIS U.S., Inc. (ARCADIS) has prepared this Remedial Investigation (RI) Work Plan (hereafter referred to as the "Work Plan") for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North, located at the former Mare Island Naval Shipyard (MINS) in Vallejo, California. The purpose of this Work Plan is to describe the field and laboratory analytical work to be performed in support of conducting an RI at the two sites. The objectives of the RI are to characterize site conditions, evaluate the nature and extent of affected soil and groundwater, refine the conceptual site model (CSM), and assess potential risk to human health and the environment. Information obtained during this process will guide future decisions made regarding DRMO South and CTA North.

DRMO South and CTA North are located within the north-central portion of MINS. DRMO South occupies land to the south of Dump Road and the DRMO site and west of Azuar Drive, while CTA North occupies the upland area to the north of Dump Road and the CTA site and west of Azuar Drive. The CTA was transferred to the City of Vallejo, which then transferred the CTA to Mare Island redeveloper, Lennar Mare Island, in 2001. Site inspections (SIs) were conducted at DRMO South and CTA North in October and November 2012 in response to a California Department of Toxic Substances Control (DTSC) request for additional information and/or data to address the possibility that affected soil and groundwater similar to that observed at the CTA may also be present on the adjacent Navy property (DTSC 2009b). The Navy also identified DRMO South, located in the vicinity of CTA North, for further investigation based on the findings of the Internal Draft Investigation Area (IA) H2 RI Report (Tetra Tech EM, Inc. 2000). IA H2 included DRMO South as well as portions of CTA North. Results of the 2012 SIs demonstrated that chemicals of potential concern (COPCs), including metals, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), total petroleum hydrocarbons as diesel (TPH-d), and total petroleum hydrocarbons as motor oil (TPH-mo), were present in soil at concentrations exceeding human health and/or ecological screening criteria and warranted additional characterization. COPCs detected in groundwater at concentrations exceeding human health and/or ecological screening criteria included metals, pesticides, PAHs, and volatile organic compounds.

The goals of the RI are to gather sufficient data to: (1) further evaluate and/or delineate COPCs identified in soil and groundwater at DRMO South and CTA North; (2) determine whether COPCs identified in soil and groundwater at the sites would result in unacceptable health risks to future receptors; and (3) update the CSM. Data obtained during the RI field work will supplement historical data gathered during previous field efforts.

The following scope of work has been defined to achieve the RI goals:

- Collection of 48 soil samples from 17 boring locations at DRMO South. Soil samples will be analyzed for metals, PAHs, PCBs, and/or total petroleum hydrocarbons-extractable (TPH-e).

- Collection of four groundwater samples from four temporary monitoring well locations at DRMO South. Groundwater samples will be analyzed for metals, PAHs, PCBs, and/or TPH-e.

- Collection of 39 soil samples from 12 boring locations at CTA North. Soil samples will be analyzed for PAHs, PCBs, TPH-e, and/or metals.

- Collection of three groundwater samples from three temporary monitoring well locations at CTA North. Groundwater samples will be analyzed for PAHs, PCBs, TPH-e, and/or metals.

Soil samples will be collected from two or more depth intervals at each boring. Soil samples will be collected from depths of up to 15 feet below ground surface (bgs) at DRMO South and up to 10 feet bgs at CTA North based on the vertical extent of impacts observed during previous investigations.

The depths of the deeper samples (i.e., samples collected from below 0.5 feet bgs) may be biased if visual observations and/or photoionization detector screening of the soil column indicate the possible presence of COPCs. Groundwater samples will be collected based on the observed depth of the water table and the final screening interval. In general, groundwater samples will be collected from the uppermost water-bearing zone.

Field work will be performed in accordance with the project-specific Sampling and Analysis Plan (included as Appendix A to this Work Plan), Waste Management Plan (included as Appendix B to this Work Plan), and Environmental Protection Plan (included as Appendix C to this Work Plan). In addition, a project-specific Site Safety and Health Plan/Accident Prevention Plan, including Activity Hazard Analyses will be prepared.

ARCADIS will prepare separate RI Reports for CTA North and DRMO South to document the results of the RI field and data analysis activities, present the results of the baseline human health and ecological risk assessments, and provide recommendations for further action, as appropriate. The human health risk assessment approach will be consistent with United States Environmental Protection Agency (USEPA) and California Environmental Protection Agency guidance as well as Navy policy. The ecological risk assessment approach will be consistent with the USEPA Ecological Risk Assessment Guidance for Superfund (1997) and the Navy Policy for Conducting Ecological Risk Assessments (Navy 1999).

The RI Reports will be certified by a California-licensed professional engineer or geologist who is actively involved in the project.

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ACRONYMS AND ABBREVIATIONS

µg/L	micrograms per liter
µg/dL	microgram per deciliter
APP/SSHP	Accident Prevention Plan/Site Safety and Health Plan
ARCADIS	ARCADIS U.S., Inc.
AUF	area use factor
AWQC	Ambient Water Quality Criteria
BAF	bioaccumulation factor
B(a)P	benzo(a)pyrene
bgs	below ground surface
BRAC	Base Realignment and Closure
CDFW	California Department of Fish and Wildlife
CDI	chronic daily intake
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
ChaduxTt	St. George Chadux and Tetra Tech EM, Inc.
CHHSL	California Human Health Screening Level
COPC	chemical of potential concern
COPEC	chemical of potential ecological concern
cPAH	carcinogenic polycyclic aromatic hydrocarbon
CSF	cancer slope factor
CSM	conceptual site model
CTA	Crane Test Area
DERP	Defense Environmental Restoration Program
DoD	Department of Defense
DQA	data quality assessment
DQO	data quality objective

DRMO	Defense Reutilization and Marketing Office
DTSC	Department of Toxic Substance Control
EcoSSL	Ecological Soil Screening Level
EDD	electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
EPC	exposure point concentration
ERA	ecological risk assessment
ESL	Environmental Screening Level
FSA	Fenced Scrapyard Area
HHRA	human health risk assessment
HI	hazard index
HQ	hazard quotient
IA	Investigation Area
IR	Installation Restoration
IR10	Installation Restoration Site 10
IR14	Installation Restoration Site 14
IRIS	Integrated Risk Information System
IW	industrial wastewater
LANL	Los Alamos National Laboratory
LOAEL	lowest observed adverse effect level
MEC	munitions and explosives of concern
mg/kg	milligrams per kilogram
MINS	Mare Island Naval Shipyard
Navy	United States Department of the Navy
NEDD	Naval Electronic Data Deliverable
NIRIS	Naval Installation Restoration Information Solution
NOAA	National Oceanic and Atmospheric Administration

NOAEL	no observed adverse effect level
NTCRA	non-time critical removal action
OEHHA	Office of Environmental Health Hazard Assessment
ORNL	Oak Ridge National Laboratory
ORP	oxidation reduction potential
PAH	polycyclic aromatic hydrocarbon
PA/SI	Preliminary Assessment/Site Inspection
PCA	Petroleum Corrective Action
PCB	polychlorinated biphenyl
PID	photoionization detector
PMO	Program Management Office
PVC	polyvinyl chloride
RAGS	Risk Assessment Guidance for Superfund
RCRA	Resource Conservation and Recovery Act
RfD	reference dose
RI	remedial investigation
RSL	Regional Screening Level
Water Board	Regional Water Quality Control Board
SAP	Sampling and Analyses Plan
SARA	Superfund Amendments and Reauthorization Act of 1986
SI	site inspection
SL	screening level
SLERA	screening level ecological risk assessment
SQuiRT	Screening Quick Reference Table
SVOC	semivolatile organic compound
SWDIV	Southwest Division Naval Facilities Engineering Command
SWMU	solid waste management unit

TPH	total petroleum hydrocarbons
TPH-d	total petroleum hydrocarbons-diesel
TPH-e	total petroleum hydrocarbons -extractable
TPH-mo	total petroleum hydrocarbons -motor oil
TRV	toxicity reference value
TtEM	Tetra Tech EM, Inc.
UCL	upper confidence limit
USC	United States Code
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
VOC	volatile organic compound
Weston	Weston Solutions, Inc.

1. INTRODUCTION

On behalf of the United States Department of the Navy (Navy), ARCADIS U.S., Inc. (ARCADIS) has prepared this Remedial Investigation (RI) Work Plan (hereafter referred to as the "Work Plan") for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North, located at the former Mare Island Naval Shipyard (MINS) in Vallejo, California. The purpose of this Work Plan is to describe the field and laboratory analytical work to be performed in support of conducting an RI at the two sites. The objectives of the RI are to characterize site conditions, evaluate the nature and extent of affected soil and groundwater, refine the conceptual site model (CSM) and assess potential risk to human health and the environment. Information obtained during this process will guide future decisions made regarding DRMO South and CTA North.

DRMO South and CTA North are located within the north-central portion of MINS (Figure 1). DRMO South occupies land to the south of Dump Road and the DRMO site and west of Azuar Drive, while CTA North occupies the upland area to the north of Dump Road and the CTA site and west of Azuar Drive. The CTA was transferred to the City of Vallejo, which then transferred the CTA to Mare Island redeveloper, Lennar Mare Island, in 2001. Site inspections (SIs) were conducted at DRMO South and CTA North in October and November 2012 in response to a California Department of Toxic Substances Control (DTSC) request for additional information and/or data to address the possibility that affected soil and groundwater similar to that observed at the CTA (on Lennar Mare Island property) may also be present on the adjacent Navy property (DTSC 2009b). The Navy also identified DRMO South, located in the vicinity of CTA North, for further investigation based on the findings of the Internal Draft Investigation Area (IA) H2 RI Report (Tetra Tech EM, Inc. [TtEM] 2000). IA H2 included DRMO South as well as portions of CTA North. Results of the 2012 SIs demonstrated that chemicals of potential concern (COPCs), including metals, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), total petroleum hydrocarbons as diesel (TPH-d), and total petroleum hydrocarbons as motor oil (TPH-mo) were present in soil at concentrations exceeding human health and/or ecological screening criteria. COPCs detected in groundwater at concentrations exceeding human health and/or ecological screening criteria included metals, pesticides, volatile organic compounds (VOCs), and PAHs.

The purpose of the RI field work, as presented in this Work Plan, is to gather sufficient data to: (1) further evaluate and/or delineate COPCs identified in soil and groundwater at DRMO South and CTA North; (2) determine whether COPCs identified in soil and groundwater at the sites would result in unacceptable health risks to future receptors; and (3) update the CSM. Data obtained during the RI field work will supplement historical data gathered during previous field efforts. Results of the RI will be used to determine whether further action is warranted at DRMO South or CTA North.

1.1 Report Organization

Including Section 1.0 Introduction, this Work Plan is organized as follows:

- Section 2.0: Site Description and Background

- Section 3.0: Conceptual Site Model
- Section 4.0: Technical Approach
- Section 5.0: Field Sampling Plan
- Section 6.0: Data Management and Review
- Section 7.0: Risk Assessment Methodology
- Section 8.0: Project Deliverables
- Section 9.0: Project Schedule
- Section 10.0: References
- Appendix A: Sampling and Analysis Plan (SAP)
- Appendix B: Waste Management Plan
- Appendix C: Environmental Protection Plan

In addition, a project-specific Accident Prevention Plan/Site Safety and Health Plan (APP/SSHP) will be developed.

1.2 Project Organization and Points of Contact

Key personnel for this project are listed below and in the SAP (Appendix A).

Navy Base Realignment and Closure (BRAC),
Program Management Office (PMO) West

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The purpose of establishing the project organization structure and points of contact is to clearly define personnel responsibilities and the lines of authority/communication to ensure

that work is completed efficiently and without deficiency in accordance with this Work Plan and performance requirements.

1.3 Regulatory Framework

The Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA) established nationwide federal programs to identify, characterize, and clean up or control contamination from hazardous waste disposal and spill sites. One of these programs, the Defense Environmental Restoration Program (DERP), codified in SARA Section 21 (10 United States Code [USC] 2701), is managed by the Department of Defense (DoD). Within the DERP, the Installation Restoration (IR) Program focuses on addressing hazardous substances, pollutants, and contaminants remaining from past activities at military installations and formerly used defense sites. Since inception of the Navy IR Program in 1981, efforts have been made to identify, assess, and remediate contaminated sites.

Investigation of DRMO South and CTA North sites is being conducted per the Navy's direction. The DoD has the authority to undertake CERCLA response actions under Title 42 USC Part 9604 (42 USC 9604), 10 USC 2708, and Federal Executive Order 12580. The Navy is the lead agency responsible for the cleanup effort, the DTSC is the lead regulatory agency overseeing IR Program activities, and the San Francisco Bay Region Regional Water Quality Control Board (Water Board) is the delegated authority for groundwater issues.

This Work Plan has been prepared pursuant to the requirements of CERCLA as amended by SARA; the National Oil and Hazardous Substances Pollution Contingency Plan (40 Code of Federal Regulations 300); and the Federal Facility Site Remediation Agreement for Mare Island in Vallejo, California, which was executed by the Navy, DTSC, and Water Board on July 15, 2002.

1.4 Community Involvement

As the lead agency, the Navy has initiated a community relations program in coordination with the DTSC. The intent of the community relations program is to solicit community input and to keep the community apprised of environmental actions at MINS. This Work Plan will be made available at the following locations as part of the community involvement process.

Administrative Record

Attn: Ms. Diane Silva, Records Manager
Naval Facilities Engineering Command Southwest
Naval Base San Diego, Building 3519
2965 Mole Road
San Diego, California 92132-5190

Point of Contact:
Ms. Diane Silva
(619) 556-1280

John F. Kennedy Library

505 Santa Clara Street
Vallejo, California 94590

(866) 572-7587

2. SITE DESCRIPTION AND BACKGROUND

2.1 Site Location and Setting

The former MINS is located on Mare Island peninsula in southwestern Solano County approximately 25 miles northeast of San Francisco. The peninsula is located within the incorporated boundaries of the City of Vallejo and is bounded by Highway 37 to the north, Carquinez Strait to the south, and the San Pablo Bay to the west (Figure 1). The Napa River (Mare Island Strait) separates Mare Island from the rest of the City of Vallejo to the east (Weston Solutions, Inc. [Weston] 2008).

DRMO South is positioned within the north-central portion of MINS, south of Dump Road and the DRMO site and west of Azuar Drive (Figures 2 and 3; Trevet 2012). CTA North is also located in the north-central portion of MINS (Figure 2). The boundaries of CTA North are roughly defined as the upland area north of Dump Road and west of Azuar Drive (Figure 4). The CTA is not included within the CTA North boundaries (Figure 2); the CTA was transferred to the City of Vallejo, which then transferred the CTA to the Mare Island redeveloper, Lennar Mare Island, in 2001.

DRMO South and CTA North were not part of the original Mare Island. These areas were situated below the water line of the San Pablo Bay until between 1911 and 1920 when the area was filled with dredge material removed from Mare Island Strait (Trevet 2012). Additional fill material composed primarily of construction debris and solid waste was deposited within a portion of the CTA North area between 1925 and 1942 (see Figure 4; Trevet 2012).

2.2 Site Description and History

Mare Island was established as a naval shipyard in 1854 following its purchase by the Navy in 1853. The primary mission of MINS was to build, maintain, and repair Navy ships and submarines. The southern portion of the island also served as a munitions storage and production facility (Trevet 2012). MINS operated for 142 years until its closure under the BRAC Program in 1996 (Trevet 2012).

2.2.1 DRMO South

DRMO South is located within the north-central portion of MINS. A large portion of the site is undeveloped while the remainder includes an industrial area and vehicle parking lots. DRMO South is currently vacant, and Section 2.3.1 presents details regarding the historical use of buildings at the site. Removal actions conducted in the neighboring DRMO site between 2005 and 2008 (non-time-critical removal action [NTCRA]) and between 2009 and 2010 (petroleum corrective action [PCA]) included the excavation of small areas of the northwest and eastern boundaries of DRMO South (Figure 3; Trevet 2012).

The NTCRA was implemented to address munitions and explosives of concern (MEC) as well as COPCs (which included metals, semivolatile organic compounds [SVOCs] including PAHs, PCBs, and pesticides) located primarily within the Fenced Scrapyard Area

(FSA; Weston 2008). The initial phase of the NTCRA involved clearing the upper 18 inches of soil from the FSA within the DRMO. However, during subsequent phases, areas with COPC concentrations exceeding the target cleanup goals, including a small area of the northwest boundary of DRMO South, were over-excavated to a maximum depth of 8.5 feet below ground surface (bgs; Weston 2008).

The PCA was implemented to remove petroleum-impacted soil from: (1) an area within and to the west of Azuar Drive; and (2) an area within and on the north and south sides of Dump Road (Figure 3; Weston 2010). Portions of the eastern boundary of DRMO South, located adjacent to Azuar Drive, were excavated to depths ranging from 17 to 20 feet bgs; and portions of the site's northern boundary, located adjacent to Dump Road, were excavated to depths ranging from 9 to 19 feet bgs. Confirmation samples indicated that concentrations of TPH as Bunker C fuel oil (C12-C40) were less than the Tier 2 screening levels (SLs) for commercial/industrial land use (i.e., 5,000 milligrams per kilogram [mg/kg] for depths between 3 and 10 feet bgs; Weston 2010).

The recent SI sampling, conducted between October and November 2012, included the collection of 51 soil samples and 11 groundwater samples from 26 borings within the DRMO South area to identify and delineate COPCs in soil and groundwater at the site. Results of the SI are summarized in Section 3.1.1. Historical data are also available from previous investigations conducted at IA H2, which included DRMO South, as summarized in the Internal Draft IA H2 RI Report (TtEM 2000).

2.2.2 CTA North

CTA North is located within the north-central portion of MINS. No buildings are or were previously located within the boundaries of CTA North (Figure 4; Trevet 2012). A majority of the site is located near a non-tidal wetland area (Figure 4).

Between 2009 and 2010, petroleum-impacted soil was excavated from an area within and immediately adjacent to CTA North as part of the PCA (Figure 4; Weston 2010). A total of 82,908 bank cubic yards of soil was removed from an approximately 3.7 acre area to depths ranging from 9 to 20 feet bgs (Weston 2010). Confirmation samples were collected and analyzed for TPH as Bunker C fuel oil (C12-C40), and the results were compared with Tier 2 SLs for commercial/ industrial land use. Excavation areas with bottom or confirmation samples exceeding the SLs were over-excavated and resampled with the exception of four sidewall samples located at the northern limit of the excavation area within CTA North (Weston 2010). These samples exhibited TPH concentrations above the Tier 2 SLs but were not over-excavated (Figure 4; Weston 2010).

The recent SI sampling, conducted between October and November 2012, included the collection of 22 soil samples and four groundwater samples from 12 borings within the CTA North area to identify and characterize COPCs in soil and groundwater at the site. Results of the SI are summarized in Section 3.1.2. Historical data are also available from previous investigations conducted at IA H2, which included portions of CTA North, as summarized in the Internal Draft IA H2 RI Report (TtEM 2000).

2.3 Potential Sources

The following sections describe potential or known sources at DRMO South and CTA North based on the results of previous environmental investigations and review of site-related documents and aerial photographs.

2.3.1 DRMO South

Information regarding historical uses of the buildings at DRMO South was presented in the Preliminary Assessment/Site Inspection (PA/SI) Work Plan (Trevet 2012). During site operation, Building 275 was designated as the "Crane Maintenance Shop" and included a paint booth. Hazardous materials (including oils and degreasers) were used and/or stored within this building. Buildings 961 and 965 were used for storage in support of Building 275 operations, and Building 831 was an open-air covered parking structure. A 500-gallon aboveground storage tank, located outside of the building perimeter on the northwest side of Building 965, supported an emergency generator. As noted in the Mare Island Health, Safety, and Environmental Building Survey Building 965, dated October 30, 1996, the out-of-service tank was drained of diesel fuel, triple-rinsed, and sent for scrap off site. No containment area related to this tank is present, and there are no visible signs of its presence or past releases. During the 2012 SI, soil boring DRMOS-SB12 was installed in the area of the former tank to characterize the location. No information was available for the other buildings located within DRMO South. Based on aerial photographs, it was determined that the open area west of Building 275 was likely used for equipment storage and vehicle parking (Trevet 2012).

Historical uses of the adjacent DRMO site included: storage of transformers, batteries, and metal scrap; storage of pesticides for maintenance purposes; bailing of paper; handling of petroleum and waste oils; and management of surplus material and scrap from MINS and other military facilities (Weston 2008). A former oil sump box, located approximately 50 feet north of Building 661 at DRMO, was determined to be the probable source of petroleum product (Bunker C fuel oil) observed along the west side of Azuar Drive. The oil sump box and associated piping were removed, and portions of Azuar Drive, including those along the eastern boundary of DRMO South, were excavated as part of the 2009-2010 PCA (see Section 2.2.1; Weston 2010). A portion of the northwestern corner of DRMO South, adjacent to Dump Road, was also excavated during the 2009-2010 PCA (see Section 2.2.1). TPH-contaminated soil in this area was attributed to intermittent and isolated disposal and releases, or backfill with undocumented contaminated materials, e.g., mixed heavy fuel oil and scrap metal (see also Section 2.3.2; CH2M HILL 2009).

Two sites identified in the Internal Draft IA H2 RI Report (TtEM 2000)—Installation Restoration Site 10 (IR10) and Installation Restoration Site 14 (IR14)—were also located in proximity to DRMO South. IR10, also known as Solid Waste Management Unit (SWMU) 107, included the portion of DRMO South located adjacent to (west of) Building 831 as well as an area to the south of the access road that bounds DRMO South to the south. The portion of IR10/SWMU 107 located south of the access road was transferred to the City of Vallejo, and the remaining portion was to be addressed as part of the CERCLA work conducted at DRMO South. Historical uses of IR10 included motor vehicle storage

and storage of electrical equipment including transformers, rectifiers, and switch gear containing PCBs (TtEM 2000). Storage of electrical equipment is not believed to have been conducted in the portion of IR10/SWMU 107 located within DRMO South.

IR14 comprised the industrial wastewater (IW) collection system that was used to collect, pretreat, and convey wastewater from various sources at Mare Island to the industrial wastewater treatment plant located on the west side of the island (TtEM 2000). The entire IW collection system at Mare Island was composed of approximately 11 IW pump stations and 26,000 linear feet of underground pipeline divided into 11 sections (IW Lines A through K) and installed at approximately 4 to 5 feet bgs. Twenty-five buildings, including an oil reclamation facility, metal cleaning and plating shops, electrical shops, photography laboratories, and paint shops were connected to the IW collection system for wastewater discharge (TtEM 2000). Pump Station 1 (Building 971), which collected wastewater from IW Line B and pumped it into IW Line A, covered an approximately half-acre area to the south of DRMO South and adjacent to Azuar Drive (TtEM 2000). Records indicate that portions of the IW pipeline were repaired over the serviceable life of the IW collection system from 1972 to 1996 (TtEM 2000). Although the exact locations of pipeline leakage and repair were not documented, the most frequent repairs were performed on the IW pipeline at its connections to the pump stations (TtEM 2000).

Potential sources of COPCs at DRMO South include:

- Vehicle (including cranes) storage, equipment storage, maintenance activities, and vehicular traffic,
- Paint booth operations,
- Hazardous materials use/storage,
- Former aboveground storage tank near Building 965,
- Land reclamation/filling activities,
- Former oil sump box and associated piping,
- COPC migration from adjoining areas including the DRMO, IR10/SWMU 107 (the former PCB storage area), and IR14 (Pump Station 1 area), and
- Bedding material in utility corridors.

2.3.2 CTA North

According to the PA/SI Work Plan, a review of site-related documents and aerial photographs did not produce any specific historical uses of CTA North (Trevet 2012). However, portions of the site are located within the 1925-1942 fill material area which is associated with the CTA site (Figure 4). Previous environmental investigations conducted at the CTA revealed elevated PAH, PCB, TPH-d, and/or metals concentrations (i.e., above

the screening values) in soil typically at depths shallower than 7 feet bgs (CH2M HILL 2010). The elevated PAH (primarily benzo[a]pyrene [B(a)P]), PCB (Aroclor 1260), and metals (arsenic and lead) concentrations were attributed to past activities conducted within the CTA, including the 1925 to 1942 fill activity, vehicle storage, operation of a classification yard and lumber salvage area, and load testing for shipyard cranes (CH2M HILL 2010).

As discussed in Section 2.2.2, four confirmation sidewall samples collected during the 2009-2010 PCA indicated that TPH was present at concentrations above the Tier 2 SLs within CTA North, at the northern limit of the excavation area along Dump Road (Weston 2010). According to the Final Technical Memorandum, Total Petroleum Hydrocarbons at the DRMO Site and Vicinity (CH2M HILL 2009), information obtained during follow-on investigations conducted in 2007 and 2008 demonstrated that the former oil sump box, located approximately 50 feet north of Building 661, was not the source of TPH found along Dump Road. Forensic chemistry findings suggested that the most probable sources of TPH contamination in the Dump Road area included intermittent and isolated disposal and releases, or backfill with undocumented contaminated materials, e.g., mixed heavy fuel oil and scrap metal (CH2M HILL 2009).

Historically, a portion of IR14 (IW) pipeline and sanitary sewer pipeline ran along Dump Road crossing through the southwest corner of CTA North. As part of the 2007 follow-on field investigation, trenches were excavated along this section of co-located IR14 pipeline and sanitary sewer line to determine whether free-phase petroleum hydrocarbons were present and whether the pipelines and their backfill could represent a preferential pathway of migration (CH2M HILL 2009). Free product and/or metal debris were observed in all four trenches at depths from 3 feet bgs to at least 9 feet bgs (CH2M HILL 2009). The southwestern corner of CTA North, including the IR14 and sanitary sewer pipeline area, was excavated as part of the 2009-2010 PCA (see Section 2.2.2). Excavation was completed to at least 10 feet bgs in this area.

In addition, a portion of storm sewer pipeline ran along Azuar Drive adjacent to the CTA and part of CTA North as well as the DRMO. According to the Final PCA Completion Report (Weston 2010), the storm sewer line parallel to Azuar Drive in this area was removed as part of the 2009-2010 PCA, and a replacement line was installed along the west side of Azuar Drive.

Potential sources at CTA North include:

- Historical activities conducted within the adjacent CTA,
- Crane storage and operation, operation of a classification yard and lumber salvage area, and vehicle storage,
- Contaminated (1925 to 1942) fill material,
- COPC migration from adjoining areas including the CTA, DRMO, and DRMO South,

- Bedding material in utility corridors, and
- IR14 (IW) and sanitary sewer pipelines.

2.4 Topography and Surface Features

The topography at both DRMO South and CTA North is relatively flat. CTA North is currently vacant and comprises primarily upland habitat. DRMO South includes both undeveloped and industrial/vehicle parking lot areas. Approximately 50 percent of the site is covered in asphalt (northeastern portion adjacent to the DRMO and Azuar Drive), while the remaining 50 percent is upland habitat.

2.5 Surface Water

No known tidal wetlands are adjacent to or within the boundaries of DRMO South or CTA North. However, non-tidal wetlands exist adjacent to both sites. Surface water is intermittent and seasonal within the non-tidal wetlands and is attributed primarily to seasonal precipitation.

2.6 Climate and Meteorology

The climate at Mare Island is moderated by its proximity to the San Francisco Bay. It is generally warm and dry in the summer and cool and wet in the winter (TtEM 2000). The average daily air temperature, as recorded at the Mare Island power plant, was 58 degrees Fahrenheit (ºF) between 1984 and 1988 (TtEM 2000). The average temperature range (minimum and maximum) for summer is 49 to 95ºF, while the average temperature range for winter is 38 to 74ºF (MINS 1996).

Daily average wind speeds measured at the Mare Island power plant were typically 5 to 10 knots (MINS 1996); and maximum velocities of 20 to 30 knots were often recorded, particularly during the winter. Winds are typically from the west and increase in velocity in the afternoon.

Average annual rainfall at Mare Island, based on daily measurements between 1878 and 1994, was 18.1 inches per year (Naval Facilities Engineering Command, Engineering Field Activity West 1995). The minimum recorded annual rainfall at Mare Island was 6.0 inches in 1976; and the maximum was 37.5 inches in 1998. Measurable precipitation typically occurs 50 to 60 days each year (TtEM 2000). In general, about 95 percent of the total precipitation occurs between October and April.

2.7 Site Geology and Hydrogeology

2.7.1 Geology

Three principal geologic units have been identified at Mare Island. These include, from top to bottom: (1) artificial fill material; (2) unconsolidated natural deposits [Younger Bay Mud]; and (3) bedrock [Battelle 2010].

Based on available boring log data, the artificial fill material at and in the vicinity of CTA North generally consists of two layers as follows:

- The upper layer of unconsolidated heterogeneous material is generally 2 to 7 feet thick and is made up of sand, gravel, silty clay, and debris in varying proportions (CH2M HILL 2010). The debris commonly consists of metal and construction debris, including brick and wood (CH2M HILL 2010). However, fragments of glass, porcelain, and textiles were also observed in borings advanced during the 2012 SI. The observed debris is likely associated with the 1925-1942 fill material.

- The lower layer primarily consists of fine-grained silts and clays, which are likely dredge spoils from Mare Island Strait that were placed in the early 1900s (CH2M HILL 2010). This layer is approximately 6 to 8 feet thick and is generally indistinguishable from the underlying unconsolidated natural deposits (CH2M HILL 2010).

The artificial fill material at and in the vicinity of DRMO South is highly heterogeneous due to land reclamation, excavation, and backfilling activities that have been conducted in this area. For the portions of DRMO South within the NTCRA or PCA areas (e.g., the northwestern corner, northeastern corner, and eastern boundary), the uppermost layer consists of clean fill material that was placed during excavation backfilling operations. In other portions of the site, the uppermost layer is generally mixed fill consisting of gravel, sand, and silt that was likely placed during land reclamation activities conducted during the 1940s (Battelle 2010). This layer occurs from the ground surface to approximately 2 to 5 feet bgs (Battelle 2010). Underlying the mixed fill layer is an approximately 4- to 7-foot thick layer consisting of fine-grained silts and clays that are most likely dredged materials placed sometime between 1911 and 1920.

Beneath the unit of artificial fill material at both CTA North and DRMO South is a layer of unconsolidated natural deposits consisting primarily of silty clay and clay with thin lenses of silty sand (CH2M HILL 2010 and Battelle 2010). This unit, commonly referred to as Younger Bay Mud, extends to at least 28 feet bgs at DRMO South (Weston 2010). The silty clay layer is predominantly composed of dark gray to olive gray clayey silt to clay with organic materials (partially decayed plant matter and wood) and brown to reddish brown peat (Trevet 2012). The organic content in the silty and clayey materials and the silt and clay content in peat varies with depth and location (Trevet 2012).

The underlying bedrock consists of steeply-dipping brown, orange, and tan arkosic sandstone, siltstone, and micaceous shale (CH2M HILL 2010 and Weston 2010). Historically, bedrock has not been encountered in the boreholes advanced at CTA North or DRMO South. However, in the adjacent CTA, the bedrock surface ranges from approximately 20 to 24 feet bgs (CH2M HILL 2010).

2.7.2 Hydrogeology

Three water-bearing zones (shallow, intermediate, and deep) have been identified at Mare Island. The shallow water-bearing zone refers to both saturated artificial fill and naturally deposited materials. In most areas of Mare Island outside of the original island footprint—

including the DRMO site, DRMO South, and CTA North—the shallow water-bearing zone is about 5 to 13 feet thick. Groundwater flow in the shallow water-bearing zone is complex and variable because of the heterogeneous nature of the artificial fill material on the island. However, groundwater flow direction generally varies from west to northwest at all three sites. The intermediate and deep water-bearing zones have not been observed in previous borings advanced at DRMO South and CTA North.

During the 2012 SI field work, the water table was generally observed at depths of 6 to 8 feet bgs at DRMO South and CTA North.

2.8 Ecological Setting

Ecological habitat at DRMO South and CTA North consists of upland habitat. Uplands are well-drained areas above the effects of tidal action (TtEM 2000). At Mare Island, uplands typically consist of grasslands and shrublands with little native vegetation remaining located along the boundary of non-tidal wetland areas (TtEM 2000). The vegetation at DRMO South and CTA North primarily includes ruderal grasses and other opportunistic plants (TtEM 2000).

Adjacent non-tidal wetland habitat contains a variety of vegetation, including cattails, pickleweed, and sedges (TtEM 2000). These areas are commonly characterized by standing water during the winter and spring and are dry during the summer and fall (TtEM 2000). The non-tidal wetland habitat is likely to support various species of invertebrates, birds, and small mammals. The upland habitat may also support some of these species.

2.9 Existing and Potential Future Land Use

Historically, DRMO South has been used for industrial activities though a large portion of the site is open space. Per the current reuse plan, future uses of DRMO South include light industrial and open space (City of Vallejo 2007). CTA North has been undeveloped open space and will remain as such per the current reuse plan (City of Vallejo 2007).

2.10 Screening Criteria

The following summarizes human health and ecological screening criteria identified in the PA/SI Work Plan for initial site screening (Trevet 2012). If the below-listed screening criteria had been updated since August 2012, when the PA/SI Work Plan was published, the most recent versions of the screening criteria were used for the data evaluation.

- Ambient concentrations of metals in fill materials at Mare Island as summarized in the Final Compilation of Technical Memoranda on Ambient Analyses of Metals in Soils and Groundwater, Mare Island, California (TtEM 2002) and Final Technical Memorandum – Ambient Fill Data Set for Molybedenum at Mare Island and Comparison to the Molybedenum Data Set for Sediment at the Non-Tidal Wetland Area of the Installation Restoration Site 17 and Building 503 Area (St. George Chadux and Tetra Tech EM, Inc. [ChaduxTt] 2012);

- United States Environmental Protection Agency (USEPA) Regional Screening Levels (RSLs; USEPA 2013) or California Human Health Screening Levels (CHHSLs; California Environmental Protection Agency [CalEPA] Office of Environmental Health Hazard Assessment [OEHHA] 2010) if an RSL was not available or not preferred for a constituent. Water Board Environmental Screening Levels (ESLs; Water Board 2013) were used for TPH constituents because neither RSLs nor CHHSLs were available;

- USEPA Interim Ecological Soil Screening Levels (EcoSSLs; USEPA 2008a);

- Oak Ridge National Laboratory (ORNL) screening levels (Efroymson et al. 1997a and 1997b);

- USEPA National Recommended Ambient Water Quality Criteria (AWQC) - Criteria Continuous Concentrations for marine environments (2009b); and

- National Oceanic and Atmospheric Administration's (NOAA's) Screening Quick Reference Tables (SQuiRTs; Buchman 2008).

In addition, background data for carcinogenic PAHs (cPAHs) in northern California surface soil were considered. The report titled Background Levels of Polycyclic Aromatic Hydrocarbons in Northern California Surface Soil (PG&E and Navy 2002) concluded that the 95th percentile of the distribution of background cPAH values was 0.92 mg/kg as B(a)P equivalents in northern California soils.

Ecological screening criteria for organic and inorganic chemicals in water were considered due to the proximity of DRMO South and CTA North to non-tidal wetland habitat. For the purpose of initial screening, it was conservatively assumed that groundwater could surface in the nearby wetlands and be available to aquatic marine species.

In addition, chemical concentrations in groundwater were conservatively screened against the residential tap water RSLs (USEPA 2013). Use of site groundwater for municipal and/or domestic supply is considered unlikely at DRMO South and CTA North. However, the most sensitive human health exposure scenario (i.e., residential exposure) is typically evaluated as part of the human health risk assessment (HHRA) to establish the baseline for unrestricted release of the property and to assess whether land use restrictions may be necessary.

Exposure assumptions and screening criteria will be refined in the future baseline HHRAs and ecological risk assessments (ERAs) to be completed for the sites (see Section 7).

3. CONCEPTUAL SITE MODEL

This preliminary CSM describes current understanding of the relationship between chemical sources, migration pathways, and exposure routes to potential receptors based on the 2012 SI results and review of site-related historical documents. This CSM will be updated and refined based on the results of the RI field work described in this Work Plan.

3.1 Nature and Extent of Affected Media

The following sections describe the nature and extent of affected soil and groundwater at DRMO South and CTA North based on the results of the 2012 SIs and previous investigations conducted at IA H2.

3.1.1 DRMO South

3.1.1.1 Soil

PCBs, petroleum hydrocarbons, PAHs, metals, pesticides, and VOCs have been detected in soil at DRMO South. Constituents detected at concentrations above the screening criteria (Table 3.1) during the 2012 SI included PCBs, TPH-d and TPH-mo, PAHs, and metals.

<u>PCBs:</u>

Results of the 2012 SI indicated that Aroclor 1260 was detected in 16 out of 51 samples at concentrations ranging from 0.021 mg/kg (estimated value) to 0.97 mg/kg (DRMOS-SB21 at 1 foot bgs; Table 3.1). The maximum detected concentration of Aroclor 1260 exceeded the 2013 USEPA industrial RSL of 0.74 mg/kg. However, the 95 percent upper confidence limit (UCL) of the mean for Aroclor 1260, calculated using USEPA ProUCL software, was 0.153 mg/kg, below both the industrial and residential RSLs. The maximum detected concentration of Aroclor 1260 was below the ecological screening level for PCBs of 40 mg/kg (ORNL screening level specific to terrestrial plants). No other Aroclors were detected in soil.

Review of the IA H2 RI data indicated that the results of historical PCB sampling conducted within the DRMO South area were generally consistent with the 2012 SI results. The exception was the relatively elevated concentration of Aroclor 1260 detected at one 1994 sample location near the site's southern boundary (Figure 3), within the portion of DRMO South that overlaps with IR10/SWMU 107 (the Building 831, former PCB storage area). A soil sample collected from 2.5 feet bgs at this location had an estimated Aroclor 1260 concentration of 2 mg/kg, above the Mare Island PCB cleanup criterion of 1 mg/kg for residential land use (TtEM 2000).

<u>Petroleum Hydrocarbons:</u>

Results of the 2012 SI indicated that TPH-d was detected at concentrations ranging from 4.2 mg/kg (estimated value) to 450 mg/kg, while TPH-mo was detected at concentrations ranging from 14 mg/kg (estimated value) to 1,600 mg/kg (Table 3.1). TPH-d and/or TPH-

mo concentrations at seven boring locations exceeded the 2013 shallow soil ESLs for residential use where groundwater is not a source of drinking water (100 mg/kg for middle distillates [diesel] and 500 mg/kg for residual fuels [motor oil]). As shown on Figure 3, these borings were located within the industrial area of the site. Reported TPH-d and TPH-mo concentrations did not exceed the 2013 shallow soil ESLs for industrial use (500 mg/kg for middle distillates and 2,500 mg/kg for residual fuels).

Review of the IA H2 RI data indicated that the results of historical TPH sampling conducted within the DRMO South area were generally consistent with the 2012 SI results. Samples with elevated TPH concentrations above the screening criteria were primarily located within the industrial areas of the site. The exception was the elevated concentrations of TPH-d (estimated 1,290 mg/kg at 0 feet bgs and estimated 1,030 mg/kg at 3 feet bgs) detected at one boring advanced in the northwestern upland area of DRMO South (Figure 3). A cluster of samples with elevated TPH-d concentrations above the residential and/or industrial ESLs at depths of up to 14.5 feet bgs was also observed in the southeastern corner of DRMO South near the Pump Station 1 area (Figure 3).

Diesel generally consists of hydrocarbons in the carbon range of C10 to C28, while motor oil varies and can include hydrocarbons with carbon numbers ranging from 18 (C18) to greater than 36 (C36+). In comparison, the product targeted during the 2009-2010 PCA (see Section 2.2.1) demonstrated a hydrocarbon pattern similar to that of Bunker C fuel oil (C12 to C40), i.e., samples contained both diesel-range hydrocarbons and 'heavier-end' hydrocarbons in the motor oil range. Note that the following hydrocarbon ranges will be specified for the analysis of TPH-d and TPH-mo, respectively, as part of the RI field work: C10 to C28 for TPH-d, and C28 to C40 for TPH-mo. Thus, these analyses will cover the carbon range analyzed for (C12 to C40) during previous confirmation sampling conducted for Bunker C fuel oil.

<u>PAHs:</u>

Results of the 2012 SI indicated that the B(a)P-equivalent concentration for total cPAHs at the site (maximum of 0.668 mg/kg in the sample collected from 7 feet bgs at DRMOS-SB20) was below the background level of 0.92 mg/kg (PG&E and Navy 2002). This suggests that PAH concentrations at DRMO South are associated with background levels in northern California surface soil. PAHs were also evaluated individually in comparison with the 2013 USEPA industrial RSLs. B(a)P was detected at concentrations above or equal to the 2013 USEPA industrial RSL of 0.21 mg/kg at eight boring locations (see Figure 3). A majority of these borings were located within the industrial area of the site; however, one boring (DRMOS-SB02) was located in the undeveloped area south of the NTCRA area boundary (Figure 3). The remaining PAHs were not detected at concentrations exceeding their respective USEPA industrial RSLs.

<u>Metals:</u>

Results of the 2012 SI indicated that arsenic was the only metal detected in soil at concentrations above the corresponding screening level benchmark for the industrial exposure scenario: the 2013 USEPA industrial RSL of 2.4 mg/kg (Table 3.1). Arsenic was

detected at concentrations ranging from 3.04 mg/kg to 57.3 mg/kg (DRMOS-SB02 at 8 feet bgs) with a 95 percent UCL of 19.3 mg/kg. The background concentration of arsenic in fill material is 36 mg/kg (TtEM 2002). Only three out of 51 detections exceeded this background value: 57.3 mg/kg (DRMOS-SB02 at 8 feet bgs), 39.3 mg/kg (DRMOS-SB03 at 8 feet bgs), and 37.2 mg/kg (DRMOS-SB11 at 7 feet bgs). The distribution of these locations (e.g., not clustered; see Figure 3) did not indicate a pattern or specific area of elevated arsenic. In addition, elevated (above background) arsenic levels were not restricted to near surface soil, indicating that the source is likely ambient concentrations of arsenic in dredge spoils used to develop the area.

Review of the IA H2 RI data indicated that lead has also been detected at DRMO South at concentrations above both its background level in fill material (59 mg/kg; TtEM 2002) and its industrial screening level benchmark (industrial CHHSL of 320 mg/kg). As shown in Figure 3, elevated lead was primarily detected in samples collected from the southeastern corner of DRMO South, near the Pump Station 1 area, at up to 13.5 feet bgs.

Based on the 2012 SI results, maximum detected concentrations of arsenic, barium, cobalt, copper, lead, manganese, nickel, selenium, and zinc exceeded both background levels and the ecological screening benchmarks (EcoSSLs specific to soil invertebrates or terrestrial plants; Table 3.1). No ecological screening criteria were available for chromium; however, the maximum detected concentration of chromium (286 mg/kg) was above the background of 140 mg/kg (TtEM 2002). This suggests that chromium concentrations in soil at DRMO South may not be attributed to background, and additional evaluation may be warranted. Chromium will be carried forward as a chemical of potential ecological concern (COPEC) for further evaluation (e.g., trophic modeling) in the baseline ERA (see also Section 7.2.1).

3.1.1.2 Groundwater

Petroleum hydrocarbons, PAHs, metals, pesticides, and VOCs have been detected in groundwater at DRMO South. Constituents detected at concentrations above the screening criteria (Table 3.1) during the 2012 SI included PAHs, metals, pesticides, and VOCs.

<u>PAHs:</u>

Results of the 2012 SI indicated that naphthalene was reported in eight groundwater samples at concentrations ranging from 0.31 micrograms per liter (µg/L, estimated value) to 0.98 µg/L (estimated value at DRMOS-SB03), above the residential tap water RSL of 0.14 µg/L. However, as discussed in Section 2.10, use of site groundwater for municipal and/or domestic supply is considered unlikely at DRMO South.

<u>Metals:</u>

Results of the 2012 SI indicated that arsenic, molybdenum, and nickel were detected in groundwater at concentrations above background (Table 3.1):

- Arsenic detections ranged from 0.976 µg/L (estimated value) to 325 µg/L (DRMOS-SB24). The background arsenic concentration in groundwater is 78 µg/L (TtEM 2002) and the drinking water RSL is 0.045 µg/L. Three of the 11 detections were greater than

the background value of 78 µg/L: 325 µg/L (DRMOS-SB24), 128 µg/L (DRMOS-SB15), and 175 µg/L (DRMOS-SB04). These locations did not correspond with the locations where arsenic was observed above background in subsurface soil (see Section 3.1.1.1).

- As shown in Table 3.1, maximum detected concentrations of molybdenum and nickel were below the residential human health screening criteria (tap water RSLs).

Based on 2012 SI results, maximum detected concentrations of arsenic and nickel were above the ecological screening benchmarks (AWQC, criteria continuous concentrations for marine environments). As discussed in Section 2.10, ecological screening criteria for inorganic chemicals in water were considered due to the proximity of DRMO South to non-tidal wetland habitat. For the purpose of initial screening, it was conservatively assumed that groundwater could surface in the nearby wetlands and be available to aquatic marine species.

Pesticides:

Results of the 2012 SI indicated that endrin was the only organochlorine pesticide detected in the groundwater. Endrin was detected in one sample (the field duplicate collected from DRMOS-SB13) at an estimated concentration of 0.026 µg/L, lower than the residential tap water RSL of 1.7 µg/L but greater than the ecological screening benchmark of 0.0023 µg/L (AWQC, criterion continuous concentration for marine environments).

VOCs:

Results of the 2012 SI indicated that chloroform was detected in one sample (DRMOS-SB11) at an estimated concentration of 1.0 µg/L, above the residential tap water RSL of 0.19 µg/L. Note that chloroform is a common laboratory contaminant when analyzing for VOCs.

3.1.2 CTA North

3.1.2.1 Soil

PCBs, petroleum hydrocarbons, PAHs, metals, pesticides, VOCs, and SVOCs have been detected in soil at CTA North. Constituents detected at concentrations above the screening criteria (Table 3.1) during the 2012 SI included PCBs, TPH-d and TPH-mo, PAHs, and metals.

PCBs:

Results of the 2012 SI indicated that Aroclor 1260 was detected in 8 out of 22 samples at concentrations ranging from 0.027 mg/kg (estimated value) to 0.39 m/kg (CTAN-SB09 at 1 foot bgs). The maximum detected concentration of Aroclor 1260 exceeded the 2013 USEPA residential RSL of 0.22 mg/kg but was below the industrial RSL of 0.74 mg/kg. The 95 percent UCL was 0.129 mg/kg, below the residential RSL of 0.22 mg/kg. The maximum detected concentration of Aroclor 1260 was well below the ecological screening

level of 40 mg/kg (ORNL screening level specific to terrestrial plants). No other Aroclors were detected in soil.

Petroleum Hydrocarbons:

Results of the 2012 SI indicated that TPH-d was detected at concentrations ranging from 5.0 mg/kg (estimated value) to 140 mg/kg, while TPH-mo was detected at concentrations ranging from 17 mg/kg (estimated value) to 5,400 mg/kg (Table 3.1). TPH-d and/or TPH-mo were detected at three boring locations (CTAN-SB03, CTAN-SB06 and CTAN-SB09; see Figure 4) at concentrations exceeding the 2013 shallow soil ESLs for residential use where groundwater is not a source of drinking water (100 mg/kg for middle distillates [diesel] and 500 mg/kg for residual fuels [motor oil]). The concentration of TPH-mo detected at CTAN-SB03 (5,400 mg/kg at 1 foot bgs) also exceeded the 2013 shallow soil ESL for industrial use of 2,500 mg/kg (residual fuels). CTAN-SB06 and CTAN-SB09 are both located within the 1925-1942 fill area. CTAN-SB03 was located near a utility corridor, and elevated TPH-d and TPH-mo concentrations above the screening criteria were only detected in the shallow soil sample (1 foot bgs) collected at this location.

Review of the IA H2 RI data indicated that the results of historical TPH sampling conducted within CTA North were generally consistent with the 2012 SI results. Samples with elevated TPH-d concentrations above the screening criteria were located within the 1925-1942 fill area (shallow soil) and near the PCA area boundary (at a depth of up to 8.5 feet bgs).

Diesel generally consists of hydrocarbons in the carbon range of C10 to C28, while motor oil varies and can include hydrocarbons with carbon numbers ranging from 18 (C18) to greater than 36 (C36+). In comparison, the product targeted during the 2009-2010 PCA (see Section 2.2.2) demonstrated a hydrocarbon pattern similar to that of Bunker C fuel oil (C12 to C40), i.e., samples contained both diesel-range hydrocarbons and 'heavier-end' hydrocarbons in the motor oil range. Note that the following hydrocarbon ranges will be specified for the analysis of TPH-d and TPH-mo, respectively, as part of the RI field work: C10 to C28 for TPH-d, and C28 to C40 for TPH-mo. Thus, these analyses will cover the carbon range analyzed for (C12 to C40) during previous confirmation sampling conducted for Bunker C fuel oil.

PAHs:

Results of the 2012 SI indicated that the B(a)P-equivalent concentration for total cPAHs at the site exceeded the background level of 0.92 mg/kg (PG&E and Navy 2002). This suggests that PAH concentrations at CTA North may not be attributable to ambient levels in northern California surface soil. PAHs were also evaluated individually in comparison with the 2013 USEPA industrial RSLs. B(a)P was detected at concentrations above or equal to the 2013 USEPA industrial RSL of 0.21 mg/kg at four boring locations (see Figure 4). Dibenzo(a,h)anthracene was also detected at one of these locations (CTAN-SB09 at 1 foot bgs) at a concentration of 0.24 mg/kg, above the industrial RSL of 0.21 mg/kg. The maximum detected concentrations of B(a)P (0.84 mg/kg and 0.68 mg/kg) occurred at

CTAN-SB09 where many of the maximum detected metals concentrations were also observed, indicating a potential source area.

Metals:

Results of the 2012 SI indicated that arsenic, lead, and mercury were the only metals that were detected at concentrations greater than the screening-level benchmark for the industrial exposure scenario:

- Arsenic detections ranged from 6.75 mg/kg to 251 mg/kg (CTAN-SB09 at 1 foot bgs). The background concentration of arsenic in fill material is 36 mg/kg (TtEM 2002). As shown in Figure 4, arsenic concentrations in four soil samples collected from two boring locations (CTAN-SB06 and CTAN-SB09, both located within the 1925-1942 fill material area) exceeded the background value.

- Lead detections ranged from 23.5 mg/kg to 23,200 mg/kg (CTAN-SB06 at 2.5 feet bgs). The background value for lead in fill material is 59 mg/kg (TtEM 2002) and the industrial CHHSL for lead is 320 mg/kg. As shown in Figure 4, lead concentrations in six soil samples collected from four boring locations (CTAN-SB05, CTAN-SB06, CTAN-SB08, and CTAN-SB09) located within the 1925-1942 fill material area exceeded the industrial CHHSL. Lead concentrations in five of the six soil samples also exceeded the sum of the industrial CHHSL plus background value, 379 mg/kg. These included the samples collected from 1 and 2.5 feet bgs at CTAN-SB06, the sample collected from 1 foot bgs at CTAN-SB08, and the samples collected from 1 and 2 feet bgs at CTAN-SB09.

- Mercury detections ranged from 0.0716 mg/kg (estimated value) to 80 mg/kg (CTAN-SB09 at 1 foot bgs). The background value for mercury in fill material is 2 mg/kg (TtEM 2002) and the industrial RSL for mercury is 43 mg/kg. The mercury concentration at one location (CTAN-SB09) located within the 1925-1942 fill material area exceeded the industrial RSL.

Based on the 2012 SI results, maximum detected concentrations of arsenic, copper, lead, manganese, mercury, molybdenum, nickel, vanadium, and zinc were above both their background levels and the ecological screening benchmarks (EcoSSLs or ORNLs). Maximum detected concentrations of barium, cobalt, and selenium exceeded the ecological screening benchmarks (EcoSSLs) but background levels were not available.

3.1.2.2 Groundwater

Petroleum hydrocarbons, PAHs, metals, and VOCs have been detected in groundwater at CTA North. Constituents detected at concentrations above the screening criteria (Table 3.1) during the 2012 SI included PAHs and metals.

PAHs:

Results of the 2012 SI indicated that the maximum and only reported concentration of naphthalene was 0.62 µg/L (estimated value) at CTAN-SB10. This detection exceeded the residential tap water RSL of 0.14 µg/L. However, as discussed in Section 2.10, use of site groundwater for municipal and/or domestic supply is considered unlikely at CTA North.

Metals:

Results of the 2012 SI indicated that only molybdenum and nickel were detected in groundwater at concentrations greater than background (Table 3.1):

- Nickel was detected at concentrations ranging from 10.6 µg/L (CTAN-SB12) to 59.2 µg/L (CTAN-SB08), above the ecological screening benchmark of 8.2 µg/L (AWQC, criterion continuous concentration for marine environments) but below the residential tap water RSL of 300 µg/L. As discussed in Section 2.10, ecological screening criteria for inorganic chemicals in water were considered due to the proximity of CTA North to non-tidal wetland habitat. For the purpose of initial screening, it was conservatively assumed that groundwater could surface in the nearby wetlands and be available to aquatic marine species.

- Molybdenum was detected at estimated concentrations ranging from 3.31 µg/L (CTAN-SB12) to 9.15 µg/L (CTAN-SB08), below both human health and ecological screening criteria.

3.2 Potential Release Mechanisms and Transport Pathways

Potential sources of COPCs identified at DRMO South and CTA North are discussed in Section 2.3.1 and 2.3.2, respectively.

Potential release mechanisms associated with these sources include:

- Past spills, leaks, or releases from hazardous materials, equipment, and/or vehicle storage and handling areas,

- Historical handling and disposal practices at Mare Island, and

- COPC migration from offsite areas;

Primary potential migration pathways include:

- Wind erosion and transport,

- Surface-water runoff erosion and transport to the non-tidal wetland area,

- Volatilization from soil or groundwater to soil vapor and the atmosphere,

- Migration to groundwater,
- Transport in groundwater, and
- Transport through preferential pathways (e.g., active or abandoned utility pipelines, more permeable utility line backfill, and the debris layer).

3.3 Potential Receptors and Exposure Pathways

Per the current reuse plan, future uses of DRMO South include light industrial and open space while CTA North is anticipated to remain as undeveloped open space (City of Vallejo 2007). Given these future use scenarios, potential receptors are limited to recreational users, construction workers (assuming some redevelopment and/or restoration of the land will occur), commercial/industrial workers (portions of DRMO South only), and ecological receptors. The most sensitive human health exposure scenario (i.e., residential exposure) is also evaluated to establish the baseline for unrestricted release of the property and assess whether land use restrictions may be necessary.

An exposure pathway is a mechanism by which receptors may come into contact with site-related chemicals. The USEPA (1989) describes a complete exposure pathway in terms of four components:

- A source and mechanism of chemical release
- A retention or transport medium
- A receptor at a point of potential exposure to a COPC
- An exposure route at the exposure point

If any of these four components is not present, then a potential exposure pathway is considered incomplete and is not evaluated further in a risk assessment. If all four components are present, a pathway is considered complete. Some pathways, although complete, can be considered insignificant if they are likely to contribute only a small fraction of the total exposure/dose.

Potentially complete exposure pathways for human receptors are discussed below and summarized on Figure 5:

- After development at the site, the surface soil may be mildly disturbed or possibly graded with subsurface soil. Commercial/industrial workers, recreational users, and resident adults and children may be exposed to surface soil (0 to 0.5 feet bgs) and subsurface soil (0 to 10 feet bgs) via incidental soil ingestion, soil particulate inhalation, and direct dermal contact.

- During development of the site, construction workers may be exposed to surface and subsurface soil via incidental soil ingestion, soil particulate inhalation, and direct dermal contact. Maintenance/utility workers performing trenching or other maintenance activities may also be exposed to surface and subsurface soil via incidental soil ingestion, soil particulate inhalation, and direct dermal contact.

- Groundwater at the site is not anticipated to be used for domestic or industrial purposes. However, to support the risk management decision process, COPCs in groundwater will be compared to applicable screening criteria for residential and industrial use.

- Due to the relatively shallow groundwater at the site, trenching activities may lead to some pooling of groundwater, which may result in exposure via direct dermal contact to construction or maintenance/utility workers. However, dewatering activities are anticipated to be implemented if groundwater does infiltrate into the trenches, minimizing the likelihood of direct contact to the trench worker.

Volatile compounds have only been detected infrequently in soil and groundwater and at relatively low concentrations. While the inhalation of volatile compounds in ambient air would likely not contribute significantly to cumulative risk at DRMO South and CTA North, it will be considered a potentially complete exposure pathway for all human receptors. Exposure to volatiles in indoor air will be evaluated if volatiles are identified in the subsurface adjacent to buildings.

Potentially complete exposure pathways for ecological receptors include wildlife and plant exposures to surface soil (0 to 2 feet bgs) via direct contact and/or ingestion, plant exposures to shallow soil (0 to depth of first groundwater or 6 feet bgs, whichever is shallower) and shallow groundwater, and ingestion of food items (i.e., plant and/or animal tissue) by wildlife. Although burrowing organisms may contact deeper soils (up to 6 feet bgs), burrowing in saturated soils does not occur. Therefore, shallow soil from 0 to 6 feet bgs will be evaluated for burrowing receptors only if the depth to groundwater is greater than 6 feet bgs. Additional detail regarding the ecological CSM is presented in Section 7.2.2.1.

Table 3.1
2012 Site Inspection Soil Data Summary
Detected Results Exceeding Screening Criteria

Former Mare Island Naval Shipyard
Vallejo, California

Analyte	Number of Detections/ Samples	Minimum Detected Concentration (mg/kg)	Maxiumum Detected Concentration (mg/kg)	*Human Health Screening Criteria*						Background Level (mg/kg)	*Ecological Screening Criteria*	
				2013 Residential RSL (mg/kg)	2013 Industrial RSL (mg/kg)	2010 CHHSL Residential (mg/kg)	2010 CHHSL Industrial (mg/kg)	2013 ESL Residential (mg/kg)	2013 ESL Industrial (mg/kg)		ORNL Screening Level (mg/kg)	EcoSSL (mg/kg)
DRMO South												
PCBs												
Aroclor 1260	16/51	0.021 J	0.97	0.22	0.74	--	--	--	--	--	40 (PCBs)	--
Petroleum Hydrocarbons												
Diesel	44/51	4.2 J	450	--	--	--	--	100	500	--	--	--
Motor Oil	49/51	14 J	1,600	--	--	--	--	500	2,500	--	--	--
PAHs												
Benzo(a)anthracene	46/51	0.0035 J	0.29	0.15	2.1	--	--	--	--	--		
Benzo(a)pyrene	45/51	0.0035 J	0.54	0.015	0.21	--	--	--	--	--	--	--
Benzo(b)fluoranthene	43/51	0.0033 J	0.4	0.15	2.1	--	--	--	--	--	--	--
Dibenzo(a,h)anthracene	3/51	0.0040 J	0.048	0.015	0.21	--	--	--	--	--	--	--
Indeno(1,2,3-c,d)pyrene	40/51	0.0031 J	0.24	0.15	2.1	--	--	--	--	--	--	--
PAHs as benzo(a)pyrene equivalent	51/51	--	--	--	--	--	--	--	--	0.92	--	--
Metals												
Arsenic	51/51	3.04	57.3	0.61	2.4	--	--	--	--	36	--	18
Barium	51/51	51	775	15,000	190,000	--	--	--	--	--	--	330
Chromium	51/51	6.95	286	120,000	1,500,000	--	--			140	--	--
Cobalt	51/51	1.81	24.1	23	300	--	--	--	--	--	--	13
Copper	51/51	9.15	386	3,100	41,000	--	--	--	--	120	--	70
Lead	51/51	6.72	301	--	--	80	320	--	--	59	--	120
Manganese	51/51	82.6	6,340	1,800	23,000	--	--	--	--	1,600	--	220
Nickel	51/51	5.57	201	1,500	20,000	--	--	--	--	130	--	38
Selenium	51/51	0.0877 J	1.31	390	5,100	--	--	--	--	--	--	0.52
Zinc	51/51	23.1	749	23,000	310,000	--	--	--	--	230	--	120
CTA North												
PCBs												
Aroclor 1260	8/22	0.027 J	0.39	0.22	0.74	--	--	--	--	--	40 (PCBs)	--
Petroleum Hydrocarbons												
Diesel	15/22	5.0 J	140	--	--	--	--	100	500	--	--	--
Motor Oil	22/22	17 J	5,400	--	--	--	--	500	2,500	--	--	--

Table 3.1
2012 Site Inspection Soil Data Summary
Detected Results Exceeding Screening Criteria

Former Mare Island Naval Shipyard
Vallejo, California

Analyte	Number of Detections/ Samples	Minimum Detected Concentration (mg/kg)	Maxiumum Detected Concentration (mg/kg)	*Human Health Screening Criteria*						Background Level (mg/kg)	*Ecological Screening Criteria*	
				2013 Residential RSL (mg/kg)	2013 Industrial RSL (mg/kg)	2010 CHHSL Residential (mg/kg)	2010 CHHSL Industrial (mg/kg)	2013 ESL Residential (mg/kg)	2013 ESL Industrial (mg/kg)		ORNL Screening Level (mg/kg)	EcoSSL (mg/kg)
PAHs												
Benzo(a)anthracene	22/22	0.0052 J	0.60	0.15	2.1	--	--	--	--	--		
Benzo(a)pyrene	22/22	0.018	0.84	0.015	0.21	--	--	--	--	--	--	--
Benzo(b)fluoranthene	22/22	0.023	1.4	0.15	2.1	--	--	--	--	--	--	--
Dibenzo(a,h)anthracene	6/22	0.014	0.24	0.015	0.21	--	--	--	--	--	--	--
Indeno(1,2,3-c,d)pyrene	22/22	0.011	0.81	0.15	2.1	--	--	--	--	--	--	--
PAHs as benzo(a)pyrene equivalent	22/22	--	--	--	--	--	--	--	--	0.92	--	--
Metals												
Antimony	22/22	0.399 J	51.3 J	31	410	--	--	--	--	8.5	--	78
Arsenic	22/22	6.75	251	0.61	2.4	--	--	--	--	36	--	18
Barium	22/22	78.3	4,100	15,000	190,000	--	--	--	--	--	--	330
Cobalt	22/22	8.4	44.6	23	300	--	--	--	--	--	--	13
Copper	22/22	50.9	24,600	3,100	41,000	--	--	--	--	120	--	70
Lead	22/22	23.5	23,200	--	--	80	320	--	--	59	--	120
Manganese	22/22	223	2,920	1,800	23,000	--	--	--	--	1,600	--	220
Mercury	22/22	0.0716 J	80	10	43	--	--	--	--	2	0.1	--
Molybdenum	22/22	0.626 J	17.8	390	5,100	--	--	--	--	4.75	2	--
Nickel	22/22	36.2	3,410	1,500	20,000	--	--	--	--	130	--	38
Selenium	22/22	0.170 J	1.23	390	5,100	--	--	--	--	--	--	0.52
Vanadium	22/22	39.9	227	390	5,100	--	--	--	--	190	2	--
Zinc	22/22	82.6	18,900	23,000	310,000	--	--	--	--	230	--	120

Notes:
CHHSL = California Human Health Screening Level
CTA = Crane Test Area
DRMO = Defense Reutilization Marketing Office
ESL = Environmental Screening Level
EcoSSL = Ecological Soil Screening Level
mg/kg = milligram per kilogram
ORNL = Oak Ridge National Laboratory
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyl
RSL = Regional Screening Level
SSL = Soil Screening Levels
-- = not applicable/not available
Laboratory results with J flags, indicating estimated values, are included.

Table 3.2
2012 Site Inspection Groundwater Data Summary
Detected Results Exceeding Screening Criteria

Former Mare Island Naval Shipyard
Vallejo, California

Analyte	Number of Detections/ Samples	Minimum Detected Concentration (µg/L)	Maxiumum Detected Concentration (µg/L)	*Human Health Screening Criteria*	Background Level (µg/L)	*Ecological Screening Criteria*	
				2013 Residential RSL (mg/kg)		AWQC (µg/L)	SQuiRT (µg/L)
DRMO South							
PAHs							
Naphthalene	8/11	0.31 J	0.98 J	0.14	--	--	--
Metals							
Arsenic	11/11	0.976 J	325	0.045	78	36	--
Molybdenum	11/11	1.28 J	22.3	78	8.8	--	23
Nickel	11/11	6.39	39.7	300	7.5	8.2	--
Pesticides							
Endrin	1/8	0.026 J	0.026 J	1.7	--	0.0023	--
Volatile Organic Compounds							
Chloroform	1/11	1.0 J	1.0 J	0.19	--	--	--
CTA North							
PAHs							
Naphthalene	1/4	0.62 J	0.62 J	0.14	--	--	--
Metals							
Molybdenum	3/4	3.31 J	9.15 J	78	8.8	--	23
Nickel	4/4	10.6	59.2	300	7.5	8.2	--

Notes:
AWQC = USEPA National Recommended Ambient Water Quality Criteria (criteria continuous concentrations for marine environments)
CTA = Crane Test Area
DRMO = Defense Reutilization Marketing Office
PAH = polycyclic aromatic hydrocarbon
RSL = Regional Screening Level
SQuiRT = National Oceanic and Atmospheric Administration Screening Quick Reference Table Criteria
-- = not applicable/not available
µg/L = microgram per liter
Laboratory results with J flags, indicating estimated values, are included.

4. TECHNICAL APPROACH

The technical approach for the RI was designed to achieve the project-specific data quality objectives (DQOs) presented in the SAP (Appendix A):

- To further evaluate and/or delineate potential COPCs identified in soil and groundwater at DRMO South and CTA North during previous investigations.

- To determine whether COPCs identified in soil and groundwater at the sites would result in unacceptable risks to human health and the environment.

Project-specific DQOs were developed using the seven step DQO development process outlined in the USEPA Guidance on Systematic Planning Using the DQO Process (USEPA 2006). Details of this process are presented in Worksheet #11 of the SAP (Appendix A).

4.1 Sampling Approach

Tables 4.1 and 4.2 present the sample depths, analytical suite, and rationale for the proposed samples at DRMO South and CTA North, respectively. In general, the locations and depths of the proposed soil and groundwater samples were chosen based on the preliminary CSM (see Section 3) to: (1) confirm and/or further delineate areas of potential COPC impacts identified through review of the 2012 SI data and historical IA H2 RI data; and (2) fill in data gap areas. At DRMO South the proposed samples include:

- Two soil samples collected from one boring location (DRMOS-SB27) near the PCA area boundary to further delineate the area of potential TPH-d impacts that is currently defined by historical sample location 02GB026. TPH-d was detected at a concentration of 3,870 mg/kg (estimated value) in the soil sample collected from 10.5 feet bgs at 02GB026.

- Four soil samples collected from two boring locations (DRMOS-SB28 and DRMOS-SB29) to delineate the PAH impacts observed at SI soil sample location DRMOS-SB02. One of the two proposed boring locations (DRMOS-SB28) was placed between DRMOS-SB02 and the NTCRA area boundary to evaluate whether the PAH concentrations observed at DRMOS-SB02 may be associated with PAHs in soil that were targeted during the NTCRA.

- Two soil samples collected from one boring location (DRMOS-SB30) to confirm the isolated area of lead and TPH-d impacts that is currently defined by historical boring location 01GB073. Elevated lead and TPH-d were detected in samples collected from 01GB073 at up to 3 feet bgs.

- Six soil samples collected from three boring locations (DRMOS-SB31 through DRMOS-SB33) to further delineate the TPH (diesel and motor oil) impacts observed at SI soil sample location DRMOS-SB04.

- Two soil samples collected from one boring location (DRMOS-SB34) near the PCA area boundary to further delineate the area of potential TPH-mo impacts that is currently defined by historical sample location DRMOGB046. TPH-mo was detected at a concentration of 5,800 mg/kg in the soil sample collected from 14 feet bgs at DRMOGB046.

- Four soil samples collected from one boring location (DRMOS-SB35) to further delineate the area of TPH (diesel and motor oil) impacts observed in the southeastern portion of the site adjacent to Azuar Drive.

- 20 soil samples and three groundwater samples collected from five boring locations (DRMOS-SB36 through DRMOS-SB40) to: (1) further delineate the area of metals [i.e., lead], Aroclor 1260, and TPH [diesel and/or motor oil] impacts to soil located in the southern portion of the site; and (2) assess the potential for COPC migration from adjoining and hydraulically upgradient areas. DRMO South is adjacent to the Pump Station 1 area and overlaps with IR10/SWMU 107 (the Building 831, former PCB storage area) to the south.

- Three soil samples collected from one boring location (DRMOS-SB41) to confirm the isolated area of TPH-d and TPH-mo impacts that is currently defined by historical sample location 10GB050. TPH-d was detected at a concentration of 11,200 mg/kg in a soil sample collected from 5.5 feet bgs at 10GB050; and TPH-mo was detected at an estimated concentration of 1,280 mg/kg in a soil sample collected from 2.5 feet bgs at 10GB050.

- Three soil samples and one groundwater sample collected from one boring location (DRMOS-SB42) to acquire additional data representative of the undeveloped/open space area.

- Two soil samples collected from one boring location (DRMOS-SB43) to confirm the isolated area of metals detections currently defined by historical sample location 01SA001.

Limited additional groundwater samples were proposed because more than six groundwater samples (11 total distributed across the site) were collected from DRMO South during the SI. A minimum of six samples is typically recommended for calculating exposure point concentrations (EPCs) for risk assessment purposes. Furthermore, the groundwater analytical results indicated that: (1) detected constituents were generally below screening criteria; and (2) detections of constituents at elevated concentrations in soil [e.g., PAHs, TPHs, etc.] did not translate to elevated concentrations of these constituents in groundwater. Groundwater samples were proposed at boring locations DRMOS-SB36, DRMOS-SB37, and DRMOS-SB39 to assess potential impacts associated with groundwater migration from hydraulically upgradient source areas (e.g., IR10/SWMU 107 [the former PCB storage area] and IR14 [Pump Station 1 area]). A groundwater sample was proposed at boring location DRMOS-SB42 because groundwater data were not collected from the open space/undeveloped portion of the site during the 2012 SI.

At CTA North, proposed samples include:

- 16 soil samples collected from four boring locations (CTAN-SB14 through CTAN-SB17) to delineate the PAH, metals (primarily lead and arsenic), and TPH impacts observed in shallow soil within the 1925-1942 fill material area (e.g., at CTAN-SB08 and CTAN-SB09). Two groundwater samples will also be collected—one from CTAN-SB14 and one from CTAN-SB16, both located downgradient of CTAN-SB09—to evaluate potential impacts to groundwater in this area.

- 12 soil samples collected from three boring locations (CTAN-SB18 through CTAN-SB20) to delineate the metals (primarily lead and arsenic) and TPH-mo impacts observed in shallow soil within the 1925-1942 fill material area (e.g., at CTAN-SB05 and CTAN-SB06). One groundwater sample will also be collected from CTAN-SB20, located hydraulically downgradient of the CTA removal area footprint, to evaluate potential impacts to groundwater in this area.

- Six soil samples collected from three boring locations (CTAN-SB21 through CTAN-SB23) to further investigate/delineate the isolated area of shallow PAH and TPH impacts that is currently defined by SI sample location CTAN-SB03. CTAN-SB21 and CTAN-SB23 will be located near the electrical utility line that runs through the northeastern portion of CTA North to determine whether the PAH and/or TPH detections at CTAN-SB03 may be associated with the utility corridor fill material. Soil samples collected from these locations will also be visually inspected for the presence of greensand (abrasive blast material).

- Two soil samples collected from one boring location (CTAN-SB24) to further delineate the area of PAH impacts defined by two SI soil sample locations (CTAN-SB12 and CTAN-SB13).

- Three soil samples collected from one boring location (CTAN-SB25) near the PCA area boundary to confirm the area of potential TPH-d impacts that is currently defined by historical sample location 01GB080. TPH-d was detected at concentrations ranging from 497 mg/kg (estimated value) to 2,640 mg/kg in soil samples collected from 2.5 to 8.5 feet bgs at 01GB080.

Note that three additional groundwater samples are proposed at CTA North because only four groundwater samples were collected from this site during the SI and these samples were not necessarily collected at or hydraulically downgradient of the most impacted sampling locations (e.g., CTAN-SB09). In addition, a minimum of six samples is usually recommended for calculating EPCs for risk assessment purposes.

4.2 Selection of Analytical Methods

Analytical methods were selected based on review of the 2012 SI data and historical IA H2 RI data as well as known COPCs at adjacent sites. Samples will be analyzed in accordance with the quality assurance/quality control protocols outlined in the SAP (Appendix A).

Soil samples collected from DRMO South and CTA North will be prepared and analyzed for one or more of the following:

- TPH extractable (TPH-e) by USEPA Method 3550B/8015B (with silica gel cleanup)
- PAHs by USEPA Method 3550B/8270C-SIM
- Metals by USEPA Method 3050B/6010B
- Mercury by USEPA Method 7471A
- PCBs by USEPA Method 3550B/8082

Groundwater samples collected from DRMO South and CTA North will be prepared and analyzed for one or more of the following:

- TPH-e by USEPA Method 3520C/8015B
- PAHs by USEPA Method 3520C/8270C-SIM
- Metals by USEPA Method 3010A/6020
- Mercury by USEPA Method 7470A
- PCBs by USEPA Method 3520C/8082

Table 4.1 Sampling Locations, Methods, and Rationale – DRMO South

Sampling Location / Identification Number	Matrix	Depth (feet bgs)	Analytical Group (Method)	Rationale
DRMOS-SB27-1	Soil	10 – 11	TPH-e (8015B)	Near the PCA excavation area boundary; further delineate the area of potential TPH-d detections above the screening criterion that is currently defined by historical sample location 02GB026, where TPH-d was detected at a concentration of 3,870 mg/kg (estimated value) at10.5 feet bgs.
-2	Soil	13 – 14		
DRMOS-SB28-1	Soil	0 – 0.5	PAHs (8270C-SIM)	Further delineate area of soil PAH detections above the screening criteria; near the NTCRA area boundary
-2	Soil	1 – 5		
DRMOS-SB29-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
DRMOS-SB30-1	Soil	0 – 0.5	TPH-e (8015B), Metals (6010/7471A)	Confirm the isolated area (defined by historical sample location 01GB073) of lead and TPH-d detections above the screening criteria.
-2	Soil	2.5 – 3.5		
DRMOS-SB31-1	Soil	0 – 0.5	TPH-e (8015B)	Further delineate the area of soil TPH-d and TPH-mo detections above the screening criteria.
-2	Soil	1 – 5		
DRMOS-SB32-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
DRMOS-SB33-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
DRMOS-SB34-1	Soil	8 – 13	TPH-e (8015B)	Near the PCA excavation area boundary; further delineate area of potential TPH-mo detections above the screening criterion that is currently defined by historical sample location DRMOGB046, where TPH-mo was detected at a concentration of 5,800 mg/kg at 14 feet bgs.
-2	Soil	13 – 15		
DRMOS-SB35-1	Soil	0 – 0.5	TPH-e (8015B)	Further delineate area of soil TPH-d and/or TPH-mo detections above the screening criteria; eastern site boundary adjacent to Azuar Drive
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 - 13		

Table 4.1 Sampling Locations, Methods, and Rationale – DRMO South

Sampling Location / Identification Number	Matrix	Depth (feet bgs)	Analytical Group (Method)	Rationale
DRMOS-SB36-1	Soil	0 – 0.5	TPH-e (8015B), PCBs (8082), Metals (6010/6020/ 7471A/7470A)	Further delineate the southern area of soil lead, Aroclor 1260, TPH-d, and/or TPH-mo detections above the screening criteria; assess the potential for constituent migration from adjoining and hydraulically upgradient source areas (e.g., IR10/SWMU 107 [the former PCB storage area] and IR14 [Pump Station 1 area]).
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 - 11		
-5	Water	TBD[ii]		
DRMOS-SB37-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 - 11		
-5	Water	TBD[ii]		
DRMOS-SB38-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 - 13		
DRMOS-SB39-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 - 13		
-5	Water	TBD[ii]		
DRMOS-SB40-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 - 11		
DRMOS-SB41-1	Soil	0 – 0.5	TPH-e (8015B)	Confirm the isolated area (defined by historical sample location 10GB050) of TPH-d and TPH-mo detections above the screening criteria.
-2	Soil	1 – 5 (from top of interval)		
-3	Soil	1 – 5 (from bottom of interval)		

Table 4.1 Sampling Locations, Methods, and Rationale – DRMO South

Sampling Location / Identification Number	Matrix	Depth (feet bgs)	Analytical Group (Method)	Rationale
DRMOS-SB42-1	Soil	0 – 0.5	TPH-e (8015B), PAHs (8270C-SIM), PCBs (8082), Metals (6010/6020/ 7471A/7470A)	Acquire additional data, including groundwater data, representative of the open space/undeveloped area to evaluate whether constituents identified at concentrations above screening criteria in the industrialized area of the site (e.g., TPH-d, TPH-mo, metals, PCBs, and PAHs) are also present in the open space/undeveloped area. Groundwater data were not collected from the open space/undeveloped area during the 2012 SI.
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Water	TBD[ii]		
DRMOS-SB43-1	Soil	0 – 0.5	Metals (6010/7471A)	Confirm the isolated area of elevated metals detections currently defined by historical sample location 01SA001.
-2	Soil	1 – 5		

bgs below ground surface
DRMO Defense Reutilization and Marketing Office
IR Installation Restoration
mg/kg milligrams per kilogram
NTCRA Non-Time Critical Removal Action
PAH polycyclic aromatic hydrocarbon
PCB polychlorinated biphenyl
PCA Petroleum Corrective Action
SI site inspection
SWMU solid waste management unit
TBD to be determined
TPH-d total petroleum hydrocarbons-diesel
TPH-e total petroleum hydrocarbons-extractable
TPH-mo total petroleum hydrocarbons-motor oil

Notes:

[i] To be collected from the capillary fringe, immediately above the water table.
[ii] To be determined based on observed depth of the water table and the final screening interval.

Table 4.2 Sampling Locations, Methods, and Rationale – CTA North

Sampling Location / Identification Number	Matrix	Depth (feet bgs)	Analytical Group (Method)	Rationale
CTAN-SB14-1	Soil	0 – 0.5	PAHs (8270C-SIM), Metals (6010/6020/7471A/7470A), TPH-e (8015B) PCBs (8082) for deep samples only (below 5 feet bgs)	Delineate the 1925-1942 shallow fill area and bound area of soil metals (e.g., lead and arsenic), TPH (diesel/motor oil), and PAH detections above the screening criteria. Obtain additional PCB data for deeper soil samples.
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 – 10 (below 1925-1942 fill layer)		
-5	Water	TBD[ii]		
CTAN-SB15-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 – 10 (below 1925-1942 fill layer)		
CTAN-SB16-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 – 10 (below 1925-1942 fill layer)		
-5	Water	TBD[ii]		
CTAN-SB17-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 – 10 (1below 1925-1942 fill layer)		
CTAN-SB18-1	Soil	0 – 0.5	Metals (6010/6020/7471A/7470A), TPH-e (8015B)	Delineate the area of soil metals (e.g., lead and arsenic) and TPH-mo detections above the screening criteria.
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 – 10 (below 1925-1942 fill layer)		
CTAN-SB19-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		

Table 4.2 Sampling Locations, Methods, and Rationale – CTA North

Sampling Location / Identification Number	Matrix	Depth (feet bgs)	Analytical Group (Method)	Rationale
-4	Soil	8 – 10 (below 1925-1942 fill layer)	Metals (6010/6020/7471A/7470A), TPH-e (8015B)	Delineate the area of soil metals (e.g., lead and arsenic) and TPH-mo detections above the screening criteria. Soil samples collected from CTAN-SB20, located near the electrical utility line, will also be visually inspected for the presence of greensand (abrasive blast material).
CTAN-SB20-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
-3	Soil	TBD[i]		
-4	Soil	8 – 10 (below 1925-1942 fill layer)		
-5	Water	TBD[ii]		
CTAN-SB21-1	Soil	0 – 0.5	TPH-e (8015B), PAHs (8270C-SIM) Metals (6010) for samples collected from CTAN-SB21 and CTAN-SB23 only	Bound the area of soil PAH, TPH-d, and TPH-mo detections above the screening criteria. CTAN-SB21 and CTAN-SB23 will be located near the electrical utility line running through the northeastern portion of CTA North to determine whether the B(a)P, TPH-mo, and/or TPH-d detections at CTAN-SB03 are associated with the utility corridor fill material. Soil samples collected from CTAN-SB21 and CTAN-SB23 will also be visually inspected for the presence of greensand (abrasive blast material).
-2	Soil	1 – 5		
CTAN-SB22-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
CTAN-SB23-1	Soil	0 – 0.5		
-2	Soil	1 – 5		
CTAN-SB24-1	Soil	0 – 0.5	PAHs (8270C-SIM)	Bound the area of soil PAH detections above the screening criteria.
-2	Soil	1 – 5		
CTAN-SB25-1	Soil	2 – 2.5	TPH-e (8015B)	Verify the relatively elevated concentrations of soil TPH-d (497 mg/kg, estimated value, to 2,640 mg/kg) detected at historical location 01GB080 from 2.5 to 8.5 feet bgs. 01GB080 is located near the PCA excavation area boundary.
-2	Soil	5 – 5.5		
-3	Soil	8 – 8.5		

bgs below ground surface
CTA Crane Test Area
mg/kg microgram per kilogram
PAH polycyclic aromatic hydrocarbon
PCA Petroleum Corrective Action
PCB polychlorinated biphenyl
TBD to be determined
TPH-d total petroleum hydrocarbons-diesel

TPH-e total petroleum hydrocarbons-extractable
TPH-mo total petroleum hydrocarbons-motor oil

Notes:

[i] To be collected from the capillary fringe, immediately above the water table.

[ii] To be determined based on observed depth of the water table and the final screening interval.

5. FIELD SAMPLING PLAN

The following section details methods and procedures for implementing the primary field activities necessary to complete the RI. These activities include biological monitoring/mitigation measures, utility clearance, site preparation, sample collection, temporary well abandonment, land survey, decontamination, investigation-derived waste management, and site restoration and demobilization. Sampling and analysis tasks are further detailed in the SAP (Appendix A).

Planned preparatory activities include notification of appropriate personnel and a field visit to identify and mark/stake proposed sample locations. A biological monitor (see Section 5.1 for further detail) will accompany the project team on the field visit to provide guidance on acceptable routes of ingress and egress. The RI field work will be coordinated with the Resident Officer in Charge of Construction and/or Caretaker Site Office representative, and at least two weeks advance notification will be provided prior to the initiation of field work.

Field personnel will be required to have read this Work Plan and the site-specific APP/SSHP prior to starting field work. In addition, prior to mobilization, ARCADIS will host a subcontractor kickoff meeting to discuss communication and coordination logistics; project goals and quality standards; and safety requirements, protocols and procedures. Daily health and safety tailgate meetings will be conducted with all on-site personnel for the duration of field work.

5.1 Biological Avoidance, Minimization, and Mitigation Measures

A qualified biological monitor approved by the United States Fish and Wildlife Service (USFWS) and the California Department of Fish and Wildlife (CDFW) will be responsible for implementing the biological avoidance, minimization, and mitigation measures required by this Work Plan. These measures will conform with those presented in the Final PA/SI Work Plan for DRMO South and CTA North (Trevet 2012) and will include the following:

- The biological monitor will have the authority to stop work if deemed necessary for any reason to protect salt marsh harvest mice or any other state or federal "Threatened or Endangered" or "Fully Protected" species, including rails, rare plants, and birds protected by the Migratory Bird Treaty Act of 1918 (16 *USC* 703-712).

- Prior to work, all contractor and subcontractor personnel will be required to attend a Biological Resource Education Program briefing conducted by the biological monitor. This briefing will: (1) review the sensitive biological resources [e.g., salt marsh harvest mouse, jurisdictional wetlands] potentially located within the work area; and (2) identify all protective measures to be implemented and complied with to ensure that these resources are not affected by work activities. New employees will attend a briefing by the biological monitor prior to participating in work activities.

- If site work is scheduled to be conducted between February 1 and September 15 (i.e., the nesting season), the biological monitor will conduct a visual inspection for nesting

birds. The visual inspection will cover all staging and storage areas, transportation routes, work areas, and soil stockpile areas, and will be completed within a reasonable time prior to the commencement of any project activities. If work is already occurring in the above-listed areas when nesting season starts, a species nesting visual inspection will not be required. If work is stopped for a longer than reasonable period of time, as determined by the qualified biologist, during the nesting season, staging and storage areas, work areas, and soil stockpile areas will be reassessed before work activities are resumed.

- The site biological monitor will inspect the work area and natural habitats occurring within a reasonable distance of the work area to identify active nests. If no active nests are found, no further mitigation will be conducted. If nesting birds are found during the visual inspections, the locations will be noted in the field log book, appropriate avoidance measures will be recommended by the biologist, and the Navy will submit the report to the USFWS and CDFW. If a qualified biologist, in coordination with the Navy, USFWS, and CDFW, determines that the project activity will not likely adversely affect the active nest, the project may proceed. The qualified biologist will monitor the activity of the nesting birds (including rails) during work activities. If the birds behave normally, the biologist will monitor them twice per week to ensure that the status has not changed. If the birds change their behavior as a result of work activities, the biologist will continue to monitor the birds as work is modified until the birds act normally. The biologist will then monitor the birds twice per week to ensure that the status has not changed.

- If a rare plant species is observed in the field, the location will be noted in the daily report, appropriate avoidance measures will be recommended by the biologist, and the Navy will submit the report to the USFWS and CDFW. If a sampling location is affected by the presence of a rare plant species, the location will be offset so as to avoid the need to remove the rare plant. If this is not feasible and the sample must be collected at that location, the Navy will notify the USFWS and CDFW, and the Navy will coordinate appropriate avoidance and mitigation measures with these agencies.

- Sample locations will be offset as necessary to avoid the removal of non-tidal wetland vegetation.

- The equipment decontamination area will be located in designated upland staging areas away from non-tidal wetlands.

- Drilling and other heavy equipment will be confined to access routes, designated staging areas, and designated drilling areas. Upland routes covered with pavement, bare ground, or non-native vegetation will be utilized as access routes to and from the designated sampling areas, to the maximum extent practicable.

- No vehicles will require access to the nontidal wetlands area, and no heavy equipment will be used within 50 feet of the nontidal wetlands.

There are no plans to collect samples from within 50 feet of the non-tidal wetlands as part of the RI. Sampling locations will remain outside of the 50-foot buffer zone for the non-tidal wetlands unless the regulatory agencies specifically request otherwise or unless extraordinary circumstances arise. If heavy equipment will be used within 50 feet of the non-tidal wetlands, the following additional mitigation measures will be implemented:

- A silt fence will be installed between the work sites and areas of non-tidal wetlands immediately following vegetation removal and before sampling activities begin to prevent the entry of salt marsh harvest mice into cleared areas. The final design and proposed location of the fencing will be reviewed and approved by the USFWS and CDFW prior to the installation. The fencing will be trenched into the ground and backfilled to prevent the mice from moving underneath the fencing. All silt fencing will be installed with stakes facing towards the work site and away from the habitat. The biological monitor will have the ability to make field adjustments to the location of the fencing depending on site-specific habitat conditions.

- The biological monitor will monitor silt fencing periodically during the work day to ensure that it remains an effective barrier to prevent entry of salt marsh harvest mice into work areas. Maintenance of the fencing will be conducted as needed throughout the work period. Any necessary repairs to the fencing will be completed within 24 hours of the initial observation of the damage. Work may continue while the fences are repaired once the qualified biologist has ensured that salt marsh harvest mice have not entered the work area.

- Each day, prior to the initiation of work within 50 feet of non-tidal wetlands, the biological monitor will thoroughly inspect the work area and adjacent habitat areas to determine if salt marsh harvest mice, rails, rare plants, or birds protected by the Migratory Bird Treaty Act are present in these areas, and will remain on site throughout the day while work activities occur in these areas. If a mouse of any species or a rail is observed, then work in the area of the observed species will not be initiated or will be stopped immediately by the biological monitor until the mouse or rail leaves the area of its own volition and the USFWS and CDFW are notified. If the mouse or rail does not leave the intrusive investigation area, work will not be reinitiated until the USFWS and CDFW are contacted and have made a decision on how to proceed with work activities. The biological monitor will direct the contractor on how to proceed accordingly.

- If vegetation removal is required within non-tidal wetlands, the vegetation will be removed under the supervision of a qualified biologist. The qualified biologist will survey the areas for salt marsh harvest mice, rails, rare plants, and birds protected by the Migratory Bird Treaty Act prior to vegetation removal. If a mouse of any species is observed within the areas being cleared of vegetation, the USFWS and CDFW will be notified. Unless otherwise approved by the USFWS and CDFW, the mouse shall be left alone and allowed to move out of the area of its own volition. Vegetation removal may only begin when no mice are observed.

- If vegetation removal is required within the 50-foot buffer zone, the process will start at the edge farthest from the non-tidal wetlands and will work its way towards the wetlands. This method will provide cover for salt marsh harvest mice and will allow them to move toward the non-tidal wetlands of their own volition as vegetation is being removed. Non-tidal wetland vegetation and any vegetation that needs to be removed for sampling activities within a 50-foot buffer zone around the non-tidal wetlands will be removed using non-motorized hand tools only (mechanized tools are acceptable). A biological monitor will be present during vegetation removal conducted within 50 feet of the non-tidal wetlands. Non-handheld equipment will be limited to the areas where the vegetation has been cut. Personnel may only walk into coastal salt marsh wetlands when accompanied by the USFWS- and CDFW-approved biologist.

5.2 Utility Clearance

ARCADIS will review available records (e.g., site plans, utility maps, as-built drawings) to screen proposed sample locations for subsurface features prior to drilling. Underground Service Alert North will be contacted at least 72 hours prior to initiating intrusive activities to locate known underground utilities in the areas where drilling is planned. In addition, a geophysical subcontractor will be retained to locate subsurface utilities using magnetic and/or electromagnetic methods. The geophysical subcontractor will mark the locations of detectable subsurface utilities and/or obstructions in and around the proposed sample locations.

5.3 Site Preparation

Prior to mobilization, a field visit will be conducted to identify and mark the locations of the proposed samples and confirm that they are accessible. The biological monitor will accompany the project team on the field visit to ensure that proposed sample locations are not within 50 feet of the non-tidal wetland. If the biological monitor determines that a proposed location is within 50 feet of the non-tidal wetland, the location will be shifted such that it is no longer within the buffer zone. The proposed sample locations will be identified using a hand-held Global Positioning System unit and marked using spray paint or stakes.

5.4 Sample Collection

5.4.1 Soil Sample Collection

The RI field work will include the collection of soil samples at various depths from 17 soil borings advanced in DRMO South and 12 soil borings advanced in CTA North (Figures 3 and 4, respectively).

Soil borings will be advanced using a direct-push (Geoprobe®) drill rig. Direct push rods will be driven in four-foot segments by a hydraulic hammer until the specified sample depth is reached. Borings will be continuously cored to depth using a dual-tube corer lined with an acetate sleeve. As the core barrel is advanced, soil will be driven into the inner acetate sleeve. After being driven four feet, the rods will be removed from the borehole,

and the acetate sleeve containing the soil removed from the sample barrel. The soil will be removed from the sleeve, its characteristics will be logged, and a sample will be collected from the desired depth interval. Lithologic descriptions will be recorded on standard boring log forms by an ARCADIS field geologist using the Unified Soil Classification System. A copy of the boring log form is provided in Attachment 1 of the SAP (Appendix A). Visual observations (e.g., for odors or staining, evidence of greensand) and photoionization detector (PID) screening of the soil column will be used to identify the possible presence of COPCs.

A dual-tube hand auger may be substituted for the Geoprobe® if access is limited or if the substitution is required by the biological monitor (e.g., to protect nesting birds). Sample locations requiring the use of a hand auger will be identified during a site walk with the field team lead and biological monitor.

Soil samples will be collected from two or more depth intervals at each boring. Soil samples will be collected from depths of up to 15 feet bgs at DRMO South and up to 10 feet bgs at CTA North.

The depths of the deeper samples (i.e., samples collected from below 0.5 feet bgs) may be biased if visual observations and/or PID screening of the soil column indicate the possible presence of COPCs.

Soil samples will be collected using clean and inert sampling tools and placed in appropriate containers provided by the analytical laboratory. Once labeled, the samples will be packaged in seam-sealing plastic bags and placed in a cooler with ice for transportation to Curtis & Tompkins Laboratories, a DoD Environmental Laboratory Accreditation Program (ELAP)-certified analytical laboratory, following standard chain-of-custody procedures. Soil samples will be analyzed for:

- Metals, including mercury, by USEPA Method 6010B/7471A,
- TPH-e by USEPA Method 8015B (with silica gel cleanup),
- PAHs by USEPA Method 8270C-SIM, and/or
- PCBs by USEPA Method 8082.

Additional details regarding the soil sampling and analytical procedures and the chain-of-custody documentation, handling, packaging, and shipping of samples are included in the SAP (Appendix A).

Following sample collection, each borehole will be properly abandoned by backfilling with bentonite grout starting from the bottom of the hole to the ground surface.

5.4.2 Well Installation and Groundwater Sample Collection

RI field work will include the installation and collection of groundwater samples from temporary groundwater monitoring wells installed at four of the boring locations (DRMOS-SB36, DRMOS-SB37, DRMOS-SB39, and DRMOS-SB42) in DRMO South and three of the boring locations (CTAN-SB14, CTAN-SB16, and CTAN-SB20) in CTA North (Figures 3 and 4).

The temporary wells will be drilled, completed, and developed by a California-licensed drilling subcontractor. The same drill rig will be used for both soil sampling and monitoring well installation in order to minimize the equipment footprint. The drilling subcontractor will work under the oversight of a field geologist who will be responsible for logging the soil column for lithology in accordance with the Unified Soil Classification System. A PID will be utilized to measure VOC levels within the work area breathing zone during drilling as well as when the soil cuttings are visibly affected.

Each well will be completed to an anticipated maximum depth of 15 feet bgs and below the water table, which is expected to be between 6 and 8 feet bgs based on SI field observations. The temporary monitoring wells will be installed using the direct-push (Geoprobe®) dual-tube system, which employs a hydraulically-operated percussion hammer (or similar) to advance an outer steel casing that contains a dual-tube liner for sampling soil. The outer casing isolates shallow layers and permits the unit to continue to probe at depth. When the borehole has been advanced to the desired depth, the monitoring well will be installed through the steel casing.

Monitoring wells will be installed to yield representative samples of the uppermost water-bearing zones (i.e., monitoring wells will be screened across the water table, and well screen length will not exceed 10 feet). The temporary monitoring wells will consist of 2-inch diameter polyvinyl chloride (PVC) slotted screen (0.010-inch) and blank riser. The screen interval will be 10 feet long with approximately 2 feet extending above the water table and 8 feet extending below the water table. Final well and well screen depths will be assessed in the field based on observations of the soil core (e.g., moisture content).

A filter pack consisting of graded silica sand (#3 sand filter pack) will be placed in the annular space as the outer rod is slowly removed, and the well casing will be surged using a vented surge block to facilitate maximum settling of the filter pack. The filter pack will extend approximately 1 to 2 feet above the top of the well screen. Alternatively, the filter pack will be installed as a pre-pack that surrounds the well screen and is emplaced with the PVC screen and riser pipe materials. Pre-pack filters will consist of an outer stainless steel mesh screen that will contain the filter pack (graded silica sand) and hold it against the inner PVC slotted screen.

A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the filter pack. Annular space remaining above the bentonite seal will be filled with a bentonite/cement grout mixture to the ground surface. Since the temporary monitoring wells will be abandoned once the field effort is finished, no surface completions will be necessary. A permanent mark (e.g., a notch) will be placed on top of the north side

of the well casing to mark the point for water level measurements. Additional details regarding the well installation procedure are provided in the SAP (Appendix A).

The newly installed wells will be developed to remove formation and filter pack materials from the wells, promote hydraulic communication with the surrounding water-bearing zone, and ensure the collection of representative groundwater samples. The bentonite/cement grout will be allowed to cure for a minimum of 24 hours, after which the wells will be developed by purging with a submersible pump or bailer until approximately three to five well casing volumes have been removed. During this process, indicator parameters (e.g., pH, temperature, and specific conductance) will be measured periodically and recorded along with observations concerning the quantity and clarity of the water withdrawn. The development will be considered complete once the indicator parameters have stabilized and the extracted water appears clear. If poor recharge rates are observed, the monitoring wells will be developed by continuously surging the well for 15 minutes and then bailing the well dry. All purge water generated during the well development activities will be containerized and disposed of in accordance with the Waste Management Plan (Appendix B).

After development, the wells will be allowed to recover for a minimum of 72 hours before sampling. Before each monitoring well is sampled, organic vapor concentrations will be measured at the top of the well using a PID and recorded on the groundwater sampling sheets. Field staff will also measure the groundwater level at each monitoring well using an electronic oil-water interface probe. If the probe indicates that separate phase product is present in the well, the thickness of the product will be measured and recorded on the groundwater sampling sheet. Separate phase product observed in the well will be removed to the extent possible during purging activities and prior to sample collection, and the groundwater sample will be collected from the approximate midpoint of the water column. If separate phase product cannot be removed during purging activities, a groundwater sample will not be collected..

Groundwater samples will be collected from the monitoring wells with a bladder pump using low-flow methodology. In accordance with the 1996 USEPA guidance document Low-Flow (Minimal Drawdown) Groundwater Sampling Procedures, the temporary wells will be purged at a rate of 500 milliliters per minute or less in order to maintain a stable depth to water with the total drawdown less than 0.3 feet (0.1 meters). Field parameters, including pH, dissolved oxygen, oxidation reduction potential (ORP), specific conductivity, temperature, and turbidity, will be monitored during the purging using a multi-parameter meter (YSI 556 or similar) equipped with an in-line flow cell and a turbidity meter. Purging will be considered complete when field parameters have stabilized over three successive readings. Stabilization criteria are as follows: ±3 percent for specific conductivity and temperature, ±10 millivolts for ORP, ±0.1 units for pH, and ±10 percent for dissolved oxygen and turbidity. If the field indicator parameters fail to stabilize, a sample will be collected after one hour of purging. If low-flow purging and sampling is not possible (e.g., due to a low well recharge rate), the well will be pumped dry and a sample collected as soon as the water volume in the well has recovered sufficiently.

Groundwater samples will be filtered in the field (prior to collection) and collected in appropriate containers provided by the analytical laboratory, packaged in seam-sealing plastic bags, and placed in a cooler with ice for transportation to the analytical laboratory (Curtis & Tompkins) following standard chain-of-custody procedures. Groundwater samples will be analyzed for:

- TPH-e by USEPA Method 8015B,
- Metals, including mercury, by USEPA Method 6020/7470A,
- PAHs by USEPA Method 8270C-SIM, and/or
- PCBs by USEPA Method 8082.

Additional details regarding the groundwater sampling and analytical procedures and the chain-of-custody documentation, handling, packaging, and shipping of samples are included in the SAP (Appendix A).

5.5 Temporary Well Abandonment

Upon completion of the sampling effort and with concurrence of the Navy, temporary monitoring wells will be abandoned in accordance with Solano County Department of Resource Management guidelines. The sampling system (i.e., PVC casing) will be removed and bentonite chips will be added via tremie pipe to each borehole and hydrated with tap water. The hydration will occur in 2-foot intervals to ensure hydration across the boring. The grout will be emplaced starting from the bottom of the hole and to the ground surface.

5.6 Land Survey

Each new monitoring well and soil boring location will be surveyed by a California-licensed land surveyor to document the horizontal positions and elevations of each location. Coordinates for each surveyed location will be established to an accuracy of 0.1 feet horizontally and 0.01 feet vertically. All horizontal coordinates will be based on California State Plan Coordinate System, North American Datum 1983, and all vertical coordinates will be based on North American Vertical Datum 1988. Top-of-casing elevations for the temporary monitoring wells will be measured at the mark/notch indicating the top of the north side of the well casing where water level measurements are taken.

5.7 Decontamination

To prevent cross-contamination, reusable equipment will be decontaminated prior to subsequent use at the sites. The following summarizes the decontamination procedures that will be followed during the field investigation:

- All heavy equipment (e.g., down-hole drilling equipment used for the collection of soil samples) will be steam-cleaned on site in a portable unit provided by the contracted

drilling company. The equipment will be cleaned until all visible soil and debris is removed.

- Soil collection and groundwater collection/purging equipment, including the associated tooling, will be cleaned after each use by following a three-stage decontamination procedure consisting of a non-phosphate detergent solution (e.g., Alconox or equivalent), a potable water rinse, and a final deionized water rinse. The sample collection equipment will be cleaned until all visible soil and debris is removed.

For equipment rinsate samples, water falling off the sampling equipment from the final rinse will be collected in appropriate sample bottles and analyzed for the same parameters as the field samples. Equipment rinsate blanks will be collected at a rate of one per day for all reusable sampling equipment.

Dedicated tubing will be used for groundwater sample collection. Further details regarding the decontamination of light and heavy equipment are provided in the SAP (Appendix A).

5.8 Investigation-Derived Waste Management and Disposal

All investigation-derived waste (i.e., excess soil from soil coring activities and soil cuttings, water from decontamination activities, purge water, etc.) will be stored in appropriately labeled, Department of Transportation-approved 55-gallon drums staged in a selected area of the Site. Open topped drums will be closed and secured with the bolt at the end of each day and when full. A representative waste sample will be collected from the waste stored in each container using a clean stainless steel trowel for soil or a clean disposable bailer for liquid waste. For soil waste, a composite sample will be collected from several areas within each drum. The waste characterization samples will be transferred into clean, laboratory-provided containers and placed in a cooler with ice for transportation to the analytical laboratory under chain-of-custody protocol. Waste characterization samples will be analyzed for the compounds required by the disposal facility. Further details of the waste handling and sampling procedures are provided in the Waste Management Plan (Appendix B).

Following waste characterization and profiling, the containers will be transferred to the waste disposal contractor for final offsite disposal at a disposal facility certified to accept Resource Conservation and Recovery Act (RCRA) or non-RCRA hazardous waste, as appropriate. The appropriate disposal facility will be determined based on the following hazardous classifications.

- Although not anticipated to be present, any soil or water classified as RCRA hazardous waste will be transported to a licensed treatment, storage, and disposal facility.

- Any soil or water classified as non-RCRA hazardous waste will be transported to a licensed hazardous waste landfill facility.

- Soil classified as nonhazardous or inert waste will be transported to a licensed landfill or soil treatment facility permitted to accept such waste. Water classified as nonhazardous will be transported to a licensed disposal facility.

A Generator Waste Profile form will be prepared and must be approved by the selected disposal facility prior to shipment. ARCADIS will prepare the profile(s) and the appropriate hazardous/non-hazardous waste manifests that will accompany each shipment for signature by the designated Navy representative(s). Two photocopies will be made of each manifest signed by the transporter; one copy will be provided to the Navy and one copy will be kept in the project file. All wastes will be transported via a licensed transporter.

5.9 Site Restoration and Demobilization

Cleanup activities will be implemented both during and upon completion of the RI field work to keep the sites and adjacent properties free from the accumulation of contractor-generated wastes. In accordance with good housekeeping and materials management practices, all waste materials, rubbish, and debris generated during the project will be placed in the appropriate containers (e.g., plastic bags or drums) as soon as practicable and disposed of properly.

Upon completion of work, all contractor vehicles and equipment will be removed from the site, and the sites and staging areas will be restored to a condition equal to that existing prior to work.

6. DATA MANAGEMENT AND REVIEW

Details of the data management and review process are provided in the SAP (Appendix A).

6.1 Data Management

The following summarizes data management tasks for field data:

- Field sampling records, including daily field logs and field forms, will be maintained in the project file as well as in a project-specific database to ensure the security and eventual distribution/reproduction of field data. All field sample results, calibrations, and calibration verifications will be recorded in the field logbook to ensure proper verification of sample results.

- A copy of the chain-of-custody forms will be faxed or e-mailed to the ARCADIS Project Chemist and/or Project Engineer on a daily basis for review and communication with the laboratory. The bottom (yellow) copy of the chain-of-custody form will be mailed or delivered to the Project Chemist or Project Engineer to be maintained in the project file.

- Field sampling data from the field records and chain-of-custody forms (e.g., date and time of sample collection, sample identification number, etc.) and survey data will be entered into the ARCADIS project database. One hundred (100) percent of manual entries into the database will be verified by checking the manual entry against the hard copy information.

The following summarizes data management tasks for laboratory data:

- Relevant raw data and documentation, including (but not limited to) logbooks, data sheets, electronic files, and final reports will be maintained by the laboratory for at least seven years. The laboratory (Curtis & Tompkins) will provide ARCADIS with 30 days advance notification before disposing of any relevant laboratory records.

- The data deliverables for this project will include USEPA Level III-equivalent and Level IV-equivalent data packages and electronic data deliverables (EDDs). The laboratory will certify that the EDDs and data reports are identical.

Project electronic data will be submitted to the Naval Installation Restoration Information Solution (NIRIS) database in the Naval EDD (NEDD) format per Environmental Work Instruction #6 (Southwest Division Naval Facilities Engineering Command [SWDIV] 2005). The contractor will identify the appropriate NEDD tables to populate and obtain approval from the Navy Remedial Project Manager to ensure completeness.

6.2 Third-Party Data Validation

An independent third-party company (Laboratory Data Consultants) will be contracted to review and validate the analytical data associated with this field investigation, with the

exception of waste characterization data. Ten (10) percent of the data will be subject to a USEPA Level IV validation, and the remaining 90 percent of the data will undergo USEPA Level III validation. The 10 percent portion of the data set will be comprised of routine field samples and field QC samples (e.g., field duplicates, field blanks, trip blanks, and equipment rinsate blanks). The validator will perform calculation checks for these data and the data for the associated QC samples (e.g., matrix spike/matrix spike duplicate, laboratory control sample, etc.). The chemical data will be validated and qualified per EWI #1 (SWDIV 2001), USEPA National Functional Guidelines for Inorganic Superfund Data Review (2010a), USEPA National Functional Guidelines for Organic Methods Data Review (2008b), as applicable. The data validation report will include the data validation findings worksheets, and the validation qualifiers will be entered electronically into the laboratory EDD.

6.3 Data Quality Assessment

After data have been reviewed by the third-party validator, the ARCADIS Project Chemist will perform a data quality assessment (DQA) to verify that the type, quality, and quantity of data collected are appropriate for their intended use. The Project Chemist will prepare a DQA report that will summarize the findings of the data assessment and discuss the usability of the data to be included in the report. Details of the DQA process are provided in the SAP (Appendix A).

7. RISK ASSESSMENT METHODOLOGY

ARCADIS will conduct human health and ecological risk assessments for DRMO South and CTA North to evaluate the potential for unacceptable risk from exposure to environmental media at these sites. The risk assessment methodology discussed in this section incorporates information presented above, including the CSM (Section 3) and the data quality and management information (Section 6), and will be used to evaluate both DRMO South and CTA North. A risk assessment report will be included in the RI Report for each site.

7.1 HHRA Methodology

An HHRA will be implemented to evaluate whether past releases of chemicals at the site have current or future potential to adversely impact human health.

The HHRA will be conducted as required by Navy guidance and policy (Navy 2001; 2008; and 2013); USEPA HHRA guidelines (USEPA 1989; 1991a, b; 2001a; 2004; and 2009a); CalEPA HHRA guidelines (DTSC 1996b; 2011c); and other guidance, literature, or site-specific information as appropriate.

7.1.1 Selection of the Dataset and COPCs

This subsection presents an overview of the data evaluation process that will be used to select the COPCs to be evaluated in the HHRA. The objectives of the data evaluation are to consider the usability of the data for the risk evaluations and to develop a list of COPCs for the site. The dataset for the HHRA will include appropriate historical data as well as data collected during the planned RI field activities. Analytical results from soil samples will be compiled in a database and evaluated for usability in the risk assessment following the Guidance for Data Usability in Risk Assessments (USEPA 1992), and according to the Risk Assessment Guidance for Superfund (RAGS), Volume 1 Part A (USEPA 1989). Estimated values (also known as “J-flagged” values) will be used at the reported concentration when conducting the statistical evaluations for the risk assessment. For duplicate samples, the higher of the two concentrations will be used in the statistical evaluation. Values reported as not detected will be evaluated using the USEPA ProUCL software’s non-detect function (USEPA 2010b). Data rejected during the data usability evaluation will not be evaluated further.

COPCs will initially be selected from the list of analytes having at least one detected value above laboratory reporting limits that has passed the data usability review. Additional screening of inorganic chemicals will include comparison with available data for ambient/background inorganics (TtEM 2002 and ChaduxTt 2012). Each organic chemical with at least one detection above the reporting limit will be considered a COPC.

7.1.2 Exposure Assessment

Selected COPCs will be evaluated in the exposure assessment.

The exposure assessment portion of the HHRA identifies potential receptor populations, the media to which they may be exposed, routes of exposure, and estimates of exposure. Potential receptors are identified based on current and future land use at the site with special consideration given to subpopulations that may be particularly sensitive to certain chemicals or that may have greater exposures. Receptors selected for the HHRA include the following (as identified in Section 3.3):

- Future commercial/industrial workers (portions of DRMO South only),
- Future recreational users,
- Future construction workers, and
- Hypothetical future residents.

Although future residential land use is not anticipated, this scenario is included to establish the baseline for unrestricted release of the property and assess whether land use restrictions may be required. If risk estimates under unrestricted land use assumptions are found to be acceptable, no land use controls would be deemed necessary to limit potential for exposure to chemical contaminants.

Information regarding the potential sources of the chemical releases, along with the previous discussion of possible receptors, form the foundation for the development of the baseline CSM. The baseline CSM will illustrate the exposure pathways to receptors potentially exposed to soil and groundwater at the site.

The following exposure scenarios will be quantitatively evaluated in the risk assessment (as discussed in Section 3.3):

- Future commercial/industrial exposure routes include incidental soil ingestion, dermal contact with soil, and inhalation of dust and vapors in ambient air.
- Future recreational exposure routes include incidental soil ingestion, dermal contact with soil, and inhalation of dust and vapors in ambient air.
- Future construction worker exposure routes potentially include incidental soil ingestion, dermal contact with soil, and inhalation of dust and vapors in ambient air. Construction workers performing work in excavations could also incidentally contact contaminated groundwater.
- Hypothetical future residential exposure routes potentially include incidental soil ingestion, dermal contact with soil, and inhalation of dust and vapors in ambient air.

Groundwater at DRMO South and CTA North is not suitable for drinking water and is not anticipated to have beneficial uses.

EPCs will be calculated for exposure media based on the identified complete exposure pathways. EPCs represent chemical concentrations in environmental media that a receptor could potentially come into contact with across an exposure area. The EPC is a purposefully conservative estimate of chemicals in media and is used to calculate chronic daily intake (CDI) of chemicals. The EPCs for aggregate risk estimation are calculated by using the best statistical estimate of an upper bound on the average exposure concentrations in accordance with USEPA guidance for statistical analysis of monitoring data (USEPA 2002a). The 95 percent UCL of the mean concentration is considered by this guidance as a conservative upper bound estimate that is not likely to underestimate the mean concentration and most likely overestimates that concentration. EPCs will be calculated for each detected constituent using the USEPA statistical program ProUCL, version 4.1.00, when the dataset is comprised of at least five detected concentrations and eight total samples. Otherwise, maximum concentrations will be used to avoid uncertainties associated with small datasets. The USEPA ProUCL software identifies the statistical distribution type (i.e., normal, lognormal, or nonparametric) for each constituent within the defined exposure area and computes the corresponding 95 percent UCL for the identified distribution type.

The amount of chemical that is taken into a person's body following exposure is referred to as CDI . CDI is expressed in units of milligrams of chemical per kilogram of body weight per day. CDI depends on the concentration of chemicals in media at the point of human contact (i.e., the EPC), and exposure assumptions specific to the receptor population, including frequency and duration of exposure, body weight, and contact rate. Default exposure assumptions from USEPA (USEPA 2012) and DTSC (DTSC 2011a) guidance will be used as appropriate. Exposure parameters for an adolescent (6 to 16 years old) will be used for the recreational receptor, and an exposure frequency of 72 days per year (3 days per week for 24 weeks) will be assumed for this receptor. Formulas used to calculate CDIs will follow USEPA RAGS (USEPA 1989) for each exposure pathway.

7.1.3 Toxicity Assessment

The purpose of the toxicity assessment is to evaluate the potential for COPCs to cause adverse effects in exposed populations. The toxicity assessment addresses both cancer risks and non-cancer health effects. The toxicity values are representative of a dose-response relationship applicable to receptor populations and are reported as either: (1) the cancer slope factor [CSF] or the inhalation unit risk for carcinogenic chemicals; or (2) the reference dose (RfD) or reference concentration for non-carcinogenic chemicals.

Toxicity values (CSF and RfD) to be used in the HHRA will be obtained from the following sources (in this order of preference):

- The Integrated Risk Information System (IRIS) database available through the USEPA Environmental Criteria and Assessments Office in Cincinnati, Ohio. IRIS, prepared and maintained by the USEPA, is an electronic database containing health risk and USEPA regulatory information on specific chemicals (http://www.epa.gov/iris/).

- USEPA Provisional Peer-Reviewed Toxicity Values, provided by the Office of Research and Development/National Center for Environmental Assessment/ Superfund Health Risk Technical Support Center, which develops these values on a chemical-specific basis when requested under the USEPA Superfund program. Provisional Peer-Reviewed Toxicity Values were obtained from USEPA Regional Screening Level tables (USEPA 2012).

- The CalEPA OEHHA's Chronic Reference Exposure Levels from December 18, 2008 (OEHHA 2008) and the Cancer Potency Values (OEHHA 2009) from July 21, 2009.

The toxicity values used in the HHRA will follow the previously listed hierarchy. For some chemicals without toxicity factors, a surrogate toxicity factor for a structurally similar chemical (when available) will be used. For example, the toxicity factor for acenaphthene will be used for acenaphthylene because no toxicity factor is available for acenaphthylene.

7.1.4 Risk Characterization

The final step in the risk assessment is the risk characterization step. For dermal and ingestion exposures, toxicity values and CDI will be combined to calculate the potential for carcinogenic risk and non-cancer health effects. For inhalation exposures, toxicity values will be combined with exposure estimated as an average exposure concentration or lifetime average exposure concentration, measured in milligrams per cubic meter (USEPA 2009a). Carcinogenic risks and non-cancer health effects will both be quantified.

The risk estimate for potential carcinogenic effects is an individual excess cancer risk. This represents the probability that an individual could have an increased risk of cancer above the background risk. An estimated risk of 1×10^{-6} means that each individual has a one in a million probability that his/her risk of incurring cancer over his/her lifetime will increase above the baseline or normal cancer rate. Estimates of cumulative excess cancer risk will be calculated as the summed products of the CSF and the CDI for each chemical and exposure pathway.

Non-cancer health effects are expressed as hazard index (HI) values, which are the sum of the hazard quotients (HQs) for individual chemicals and exposure pathways. HQs will be calculated as the chemical-specific CDI to the RfD. Where appropriate, non-cancerous HQs will be added together for chemicals that affect the same target organs to calculate a target organ HI.

Potential adverse health effects from lead concentrations will be evaluated using methods different from those conventionally used for other carcinogens and non-carcinogens. Adverse health effects resulting from the uptake of lead are evaluated using available USEPA and DTSC models. For resident children, the DTSC (2011b) Lead Spread 8 model calculates blood lead levels from exposure to soil lead concentrations in addition to other routes. The CalEPA's OEHHA developed a new toxicity evaluation of lead replacing the 10 microgram per deciliter (µg/dL) threshold blood concentration with a source-specific "benchmark change" of 1 µg/dL. One µg/dL is the estimated incremental increase in children's blood lead that would reduce intelligence quotient by up to one point (DTSC

2011b). For adults, the DTSC (2011b) recommends use of their modified version of the USEPA Adult Lead Model, which includes the benchmark change criterion.

For the purposes of the RI, the potential for unacceptable human health risk will be identified by using the following risk thresholds:

- In interpreting estimates of excess lifetime cancer risks, action is generally considered to be warranted when the multi-chemical aggregate cancer risk for all exposure routes within a specific exposure scenario exceeds one in ten thousand (1×10^{-4}) under USEPA oversight or one in a million (1×10^{-6}) under DTSC oversight. In general, action is not required for risks falling below the estimated cancer risk of 10^{-6}.

- Under both USEPA and state guidance, an unacceptable non-cancer hazard exists if the multi-chemical aggregate non-cancer hazard for all exposure routes within a specific exposure scenario exceeds a target non-cancer HI of 1.

- If lead concentrations in environmental media result in a predicted blood-lead level of 10 micrograms per deciliter in greater than 5 percent of the potentially exposed population, lead is present at unacceptable levels.

7.2 Ecological Risk Assessment Methodology

An ERA is the process of estimating and characterizing the likelihood that adverse ecological effects are occurring or have occurred as a result of exposure to one or more chemical stressors (USEPA 1997). A screening level ERA (SLERA) will be conducted as the first step as recommended by Navy policy for conducting ERAs (Navy 1999 and 2012) and consistent with USEPA guidance (USEPA 1997; 1998; 1999b; and 2001b) and DTSC (1996) guidance and subsequent EcoNotes (DTSC 1998; 1999b; 2000; 2002; and 2009a). The results of the SLERA will be used to determine whether further evaluation is needed to meet site remedial objectives. Further evaluation includes additional phases of the risk assessment process which are more detailed and site-specific than the preceding phase. Each phase is briefly described below:

- **Tier 1 SLERA** – The Tier 1 SLERA represents Steps 1 and 2 of the Navy policy (Navy 1999 and 2012). It is analogous to Steps 1 and 2 of the Superfund guidance (USEPA 1997). The Tier 1 SLERA employs existing data and conservative assumptions regarding contaminant exposure to develop a screening-level problem formulation ecological conceptual model, exposure evaluation, toxicity evaluation, and risk calculations. Potential for unacceptable ecological risk is indicated by constituent concentrations exceeding receptor- and media-specific screening levels.

- **Tier 2 Baseline ERA** – The Tier 2 Baseline ERA consists of several steps (Steps 3 through 7) designed to refine exposure and risk estimates in a scientifically defensible manner. Steps may include use of less-conservative exposure assumptions (Step 3a) for the Tier I SLERA or additional evaluation and/or collection of data (Steps 3b through 7). Potential for unacceptable risk is indicated by HQs greater than 1.

- **Tier 3 Evaluation of Remedial Alternatives** – Tier 3 is initiated when the results of the Tier 2 Baseline ERA indicate that site-related chemicals of ecological concern pose unacceptable risks to one or more assessment endpoints. Remedial alternatives, including a no-action alternative, are developed and evaluated with the goal of reducing ecological risks to an acceptable level. A preferred remedy is selected based on effectiveness in reducing risks, potential ecological impacts related to the remedy, and residual risks. The preferred remedy also will be selected to balance both human health and ecological concerns.

At the conclusion of each phase (or tier), a risk management decision is made regarding site status. The decision criteria for the SLERA include the following three possible outcomes for the sites:

- The site does not pose an unacceptable risk and no further action is warranted.

- The site poses a potentially unacceptable risk that requires additional evaluation through a Tier 2 Baseline ERA.

- The site poses a potentially unacceptable risk and accelerated site remediation is warranted. The evaluation of remedial alternatives is completed in a Tier 3 evaluation.

The ERAs for DRMO South and CTA North will include Tier 1 SLERAs. Analyte/receptor pairs that demonstrate potential risks based on the Tier 1 SLERA may be carried forward and evaluated in the Tier 2 assessment (Step 3a). The Tier 2 Baseline ERA is based on predictive risk calculations (i.e., the HQ method), which compare a conservative exposure estimate (e.g., dose for higher level organisms or exposure concentration in environmental media for ecological communities) to a toxicity reference value (TRV) for the selected target receptor. Doses will be calculated using refined site-specific exposure assumptions, as appropriate. The use of refined exposure assumptions is specified in Step 3a so that the risk characterization results are not overly conservative due to the use of generic exposure assumptions (used in the Tier I SLERA).

7.2.1 Selection of the COPECs

COPECs are constituents that are likely site-related and frequently detected in site media at concentrations that may pose a risk to the environment. Constituents detected in site media may not be selected as COPECs if they are essential nutrients typically found at relatively high concentrations in site media (e.g., calcium, magnesium, potassium, and sodium), are known laboratory contaminants and are found at elevated concentrations in laboratory blank samples, or are naturally occurring compounds detected in site media at concentrations comparable to local background or ambient concentrations. Ambient/background concentrations of metals are available for Mare Island (TtEM 2002; ChaduxTt 2012). Alternatively, site-specific background values may be developed, if appropriate.

For the purpose of the ERA, each constituent detected above its respective laboratory reporting limit at a concentration greater than its ambient/background level, as applicable,

will be selected as a COPEC. Chemicals that are not detected or analyzed will be considered not present in site media and will not be quantitatively evaluated. Uncertainties related to this assumption will be discussed in the uncertainty section of the ERA.

7.2.2 Exposure Assessment

The exposure assessment describes the potential or actual contact of chemicals with receptors. The objective of the exposure assessment is to provide an exposure profile that identifies receptors and exposure pathways to be evaluated, and describes the intensity and spatial and temporal extent of contact of receptors to COPECs. Complete exposure pathways and receptors evaluated in the ERA will be consistent with the CSM as discussed in Section 3 and developed for ecological receptors below.

7.2.2.1 Ecological CSM

The DRMO South open space area and CTA North area consist of upland habitats characterized by open grasslands and shrublands with little native vegetation. The DRMO South industrial area does not currently contain ecological habitat. This area is expected to remain in-use for light industrial purposes and does not require evaluation in the ERA as no complete exposure pathways are present. Non-tidal wetlands are located adjacent to the upland areas of DRMO South and CTA North.

As discussed in Section 3.3, potentially complete exposure pathways for ecological receptors include wildlife and plant exposures to surface soil (0 to 2 feet bgs) via direct contact and/or ingestion, plant exposures to shallow soil (0 to 6 feet bgs) and shallow groundwater, and ingestion of food items (i.e., plant and/or animal tissue) by wildlife. Although burrowing organisms may contact deeper soils (up to 6 feet bgs), burrowing in saturated soils does not occur. Therefore, shallow soil from 0 to 6 feet bgs will be evaluated for burrowing receptors only if the depth to groundwater is greater than 6 feet bgs.

<u>Tier 1:</u>

Receptor groups selected for the upland habitat in the Tier I SLERA include the ecological community (terrestrial plants and soil invertebrates) and wildlife (birds and mammals). The aquatic community in the adjacent non-tidal wetland areas will also be evaluated for aquatic plants, benthic invertebrates, birds, and mammals.

<u>Tier 2:</u>

If Tier 2 Baseline ERA calculations are necessary, up to five vertebrate terrestrial receptors will be used for food-chain analysis to evaluate risk to passerine birds, raptors, small mammals, and large carnivorous mammals, as necessary, based on the COPEC/receptor group pairs that exceed the Tier I screening criteria:

- California vole (Microtus californicus)
- Ornate shrew (*Sorex ornatus*)

- Western meadowlark (*Sturnella neglecta*)
- Northern harrier (*Circus cyaneus*)
- Gray fox (Urocyon cinereoargenteus)

Additionally, four vertebrate species (three birds and one mammal) may be evaluated (as necessary) as representative non-tidal wetland habitat receptors for food-chain analysis to evaluate risks to small mammals, shorebirds, waterfowl, and wading birds:

- Salt marsh harvest mouse (*Reithrodontomys raviventris*)
- Killdeer (Charadrius vociferous)
- Mallard (Anas platyrhynchos)
- Great Blue Heron (*Ardea herodias*)

7.2.2.2 Exposure Parameters

For the Tier I SLERA, screening values protective of each receptor group will be selected from the literature. The assumptions incorporated into these screening levels differ based on the source of the screening levels (see Section 7.2.3) but are appropriate to conservatively identify if potential risk may be associated with exposure to site media.

If Tier 2 Baseline ERA calculations are necessary, exposure assumptions from the literature will be used for soil and prey ingestion rates, body weight, dietary composition, and home range. Standard exposure assumptions for these parameters are available from the USEPA (1993) and ORNL (Sample et al. 1996).

For food chain modeling, bioaccumulation of chemicals will be evaluated using bioaccumulation factors (BAFs) that estimate concentrations of COPECs in biota tissue from soil. BAFs available in the literature (USEPA 1999a; Bechtel-Jacobs 1998; and Sample et al. 1998a,b) can be simple multipliers or regression models. Chemicals with low octanol-water partitioning coefficient (log K_{ow}) values generally do not bioaccumulate (DTSC 1996a; USEPA 2000b). Only COPECs with the potential to bioaccumulate, namely some metals and organics with log K_{ow} values greater than 3.5 (USEPA 2000b), will be evaluated.

7.2.2.3 Exposure Point Concentrations

EPCs will be developed using appropriate historical data as well as data collected during the planned RI field activities that meet the data useability requirements described in Section 6 and Appendix A. Rejected data (R or UR data qualifiers) will not be included in the risk assessment datasets.

For the Tier I SLERA, maximum concentrations in soil in the DRMO South open space and CTA North areas will be used for screening purposes. Shallow soil (0 to 6 feet bgs) EPCs will be used if the depth to groundwater is greater than 2 feet bgs; otherwise maximum concentrations in surface soil (0 to 2 feet bgs) will be used. Because no sediment data are available from the nearby non-tidal wetlands, surface soil EPCs for the upland areas will conservatively be used to evaluate potential impacts to aquatic receptors assuming that these surface soils have the potential to migrate to the nearby wetlands.

If Tier 2 Baseline ERA calculations are necessary, EPCs will be based on the 95 percent UCL of the mean concentration consistent with USEPA and CalEPA guidance. The EPCs will be calculated over the exposure areas selected for the Tier 2 Baseline ERA (see Section 7.2.2.4). The 95 percent UCL concentrations will be calculated using ProUCL software, version 4.1.00, when the dataset is comprised of at least five detected concentrations and eight total samples. Otherwise, maximum concentrations will be used to avoid uncertainties associated with small datasets.

7.2.2.4 Exposure Areas

Future land use is assumed to remain as open space. Accordingly, for the Tier I SLERA, the DRMO South open space and CTA North areas will be screened individually using maximum detected concentrations in each area.

If Tier 2 Baseline ERA calculations are necessary, exposure areas based on the home range of each wildlife receptor will be used if the home ranges are smaller than the DRMO South open space and CTA North areas. For receptors with larger home ranges, area use factors (AUFs) may be applied to account for use of other nearby habitat. For the ecological community, exposure areas protective of populations of these receptors will be selected.

7.2.3 Effects Assessment

The effects assessment includes the identification and development of toxicity values for ecological receptors. Following CalEPA guidance (DTSC 1996a) toxicity values will be based on “no observed adverse effect levels” (NOAELs). The NOAEL is the concentration/dose at or below which no adverse effects on the test organism are observed. To evaluate a range of risk estimates for ecological receptors, “lowest observed adverse effect levels” (LOAELs) or alternative “upper bound” toxicity values will also be used.

Evaluation of risks associated with ambient/background conditions will also be provided for comparison with estimated site risks.

<u>Tier 1:</u>

For the Tier 1 SLERA, ecological risk-based screening levels appropriate for each terrestrial receptor group will be selected according to the following hierarchy:

- 2008 USEPA EcoSSLs

- ORNL soil screening levels (Efroymson et al. 1997a,b)
- USEPA Region 5 (USEPA 2003) RCRA Ecological Screening Levels for soil
- Los Alamos National Laboratory (LANL) Ecological Screening Levels from the EcoRisk Database, version 3.1

For the evaluation of wetlands, sediment screening levels will be used from the following sources:

- Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystem (MacDonald et al. 2000);
- ORNL: Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota (Jones et al. 1997)
- USEPA Supplemental Guidance to RAGS: Region 4 Bulletins, Ecological Risk Assessment (USEPA 2001c)

The upland criteria will be applied to wetlands if criteria are not available for a chemical, and vice versa.

COPEC/receptor pairs exceeding risk-based screening levels will be retained for additional evaluation through a Tier 2 Baseline ERA. COPEC/receptor pairs with no screening values will be discussed in the uncertainty section of the ERA.

Tier 2:

For target receptors or communities that are exposed directly to the media in which they live (e.g., plants and invertebrates), exposure is expressed in terms of measured concentrations of constituents in the media. For example, the concentrations of constituents in soil are used directly to estimate the intake received by terrestrial plants. Toxicity values based on media concentrations (i.e., soil concentrations) will be used for vegetation and soil-dwelling organisms, and will be the same as the screening values used for the Tier I SLERA.

For wildlife, exposure doses in units of milligrams per kilogram body weight per day calculated from food chain modeling are compared to TRVs. A TRV is defined as a daily dose of a chemical expressed in milligrams per kilogram body weight per day, and represents a dose associated with the NOAEL or LOAEL at the population level of ecological organization even if exposure occurs over an extended duration. As wildlife TRVs are route-specific, they are used to evaluate effects from exposure via specific pathways (e.g., ingestion pathways). TRVs will be selected from the following sources in order of preference:

- Low and high TRVs developed by the Navy - USEPA Region 9 Biological Technical Assessment Group (USEPA 2002b)

- 2008 USEPA EcoSSLs
- Toxicological Benchmarks for Wildlife (Sample et al. 1996)
- Screening Level Ecological Risk Assessment Protocol, Appendix E (USEPA 1999a)
- Supplemental Guidance to RAGS: Region 4 Bulletins, Ecological Risk Assessment (USEPA 2001c);
- LANL Ecological Screening Levels from the EcoRisk Database, version 3.1

Both NOAEL-based and LOAEL-based TRVs will be used to calculate HQs for each target receptor. The NOAEL TRV is consistent with a chronic no-effect level; the no-effect level is the highest dose at which no effect to the test organism was observed. The LOAEL TRV is consistent with the LOAEL. Consistent with Biological Technical Assistance Group (DTSC 2000; 2002) and USEPA (1997) guidance, adverse population-level effect endpoints are considered to be based on survival, growth, and reproduction. Differences in body weight between the site-specific wildlife receptors and laboratory animals used in the studies to develop the TRVs are not considered significant (DTSC1999a). Therefore, scaling factors will not be used to develop TRVs.

7.2.4 Risk Characterization

Risk characterization integrates the results of the exposure assessment and effects assessment and is subject to the uncertainties associated with both of these assessments as well as with the problem formulation step. Risk characterization includes estimation of risks (i.e., HQs) and evaluation of additional lines of evidence important for interpreting the risk results.

For the ERA, COPECs will be considered unlikely to pose an unacceptable risk to a receptor group if the HQ based on the NOAEL TRV does not exceed one. Adverse effects will be considered possible but unlikely for COPECs if the HQ based on the NOAEL TRV exceeds one but the HQ based on the LOAEL TRV does not exceed one. COPECs will be considered likely to pose an unacceptable risk if the HQ based on the LOAEL TRV exceeds one. For special-status species, protection is at the level of the individual, and, therefore, unacceptable risks are considered probable if the HQ based on the NOAEL TRV exceeds one.

8. PROJECT DELIVERABLES

ARCADIS will prepare separate RI Reports for CTA North and DRMO South to document the results of the RI field and data analysis activities, present the results of the human health and ecological risk assessments, and provide recommendations for further action, as appropriate. The RI Reports will be certified by a California-licensed professional engineer or geologist who is actively involved in the project.

Each RI Report will include the following elements, at a minimum, as recommended in the USEPA Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA (1988):

- Introduction, including the project and site background (site description, site history, summary of previous investigations);
- Study area investigation, including site assessment methodology;
- Physical characteristics of the study area;
- Nature and extent of the potentially identified COPCs, including descriptions and results of the RI activities;
- COPCs fate and transport, including potential routes of migration;
- Baseline risk assessments, including descriptions and results of the human health and ecological risk assessments; and
- Summary, conclusions, and recommendations.

9. PROJECT SCHEDULE

The anticipated schedule for implementation of the RI is provided as Figure 5. Mobilization is scheduled for November 2013, and it is anticipated that it will take on the order of one month to complete the field effort. However, this schedule may be modified based on regulatory approvals, field conditions, and site access.

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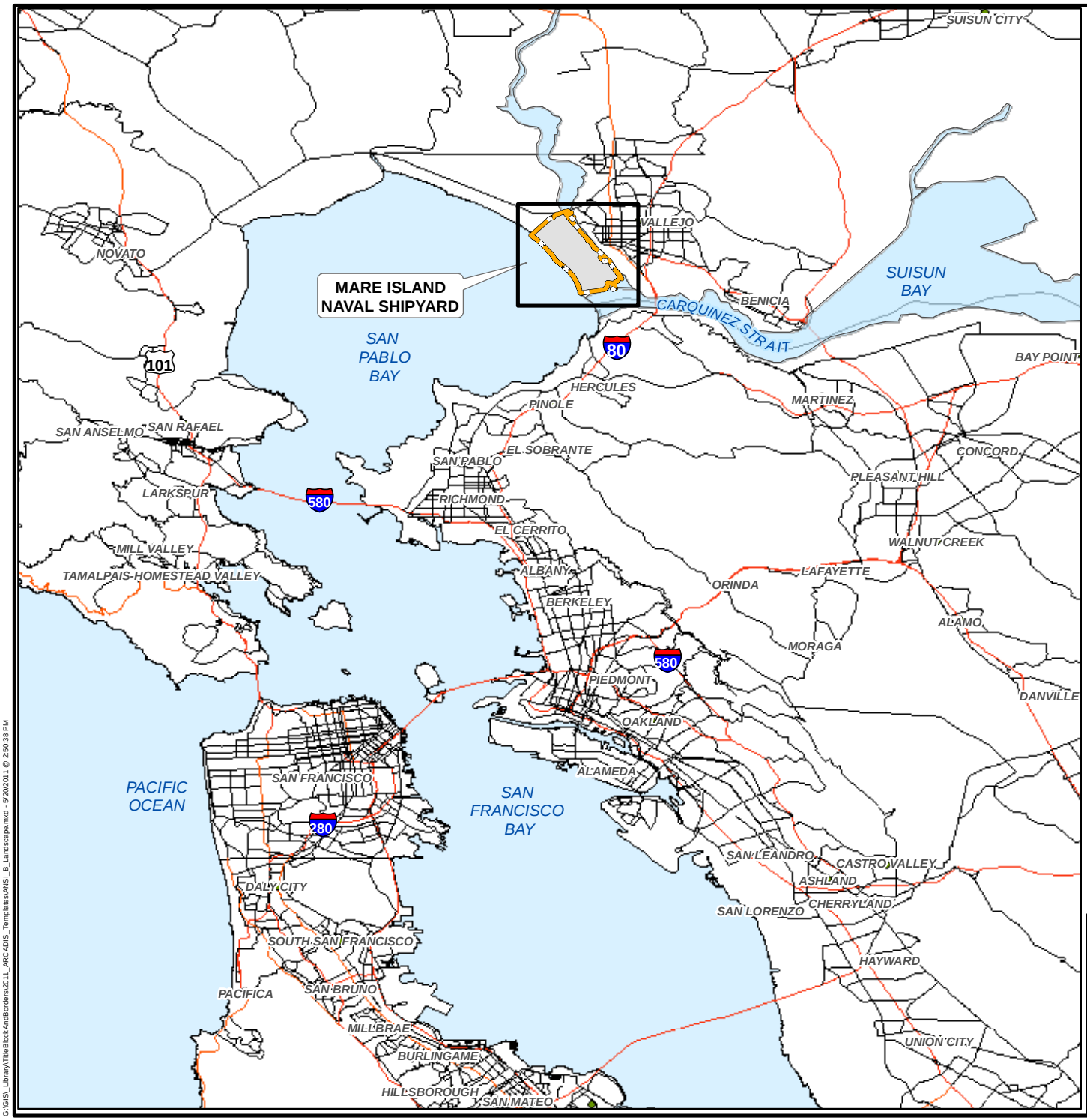
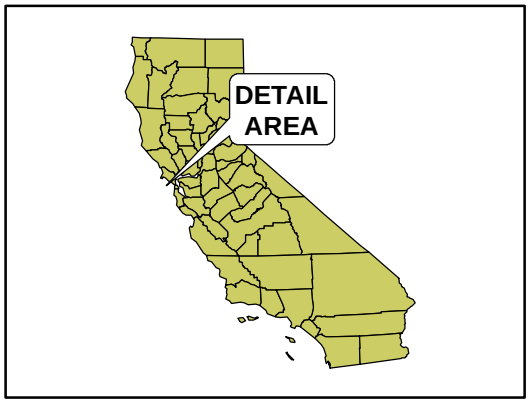
Figures





LEGEND:

MARE ISLAND NAVAL SHIPYARD BOUNDARY

SOURCE: ENVIRONMENTAL SYSTEMS RESEARCH INSTITUTE (ESRI) USA SPATIAL FEATURE CLASSES

0 7 14
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MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

REGIONAL MAP

ARCADIS

FIGURE
1

CITY: SAN FRANCISCO DIV/GROUP: ENV/GIS DB: User: MEstifanos LD: PIC: PM: TM:
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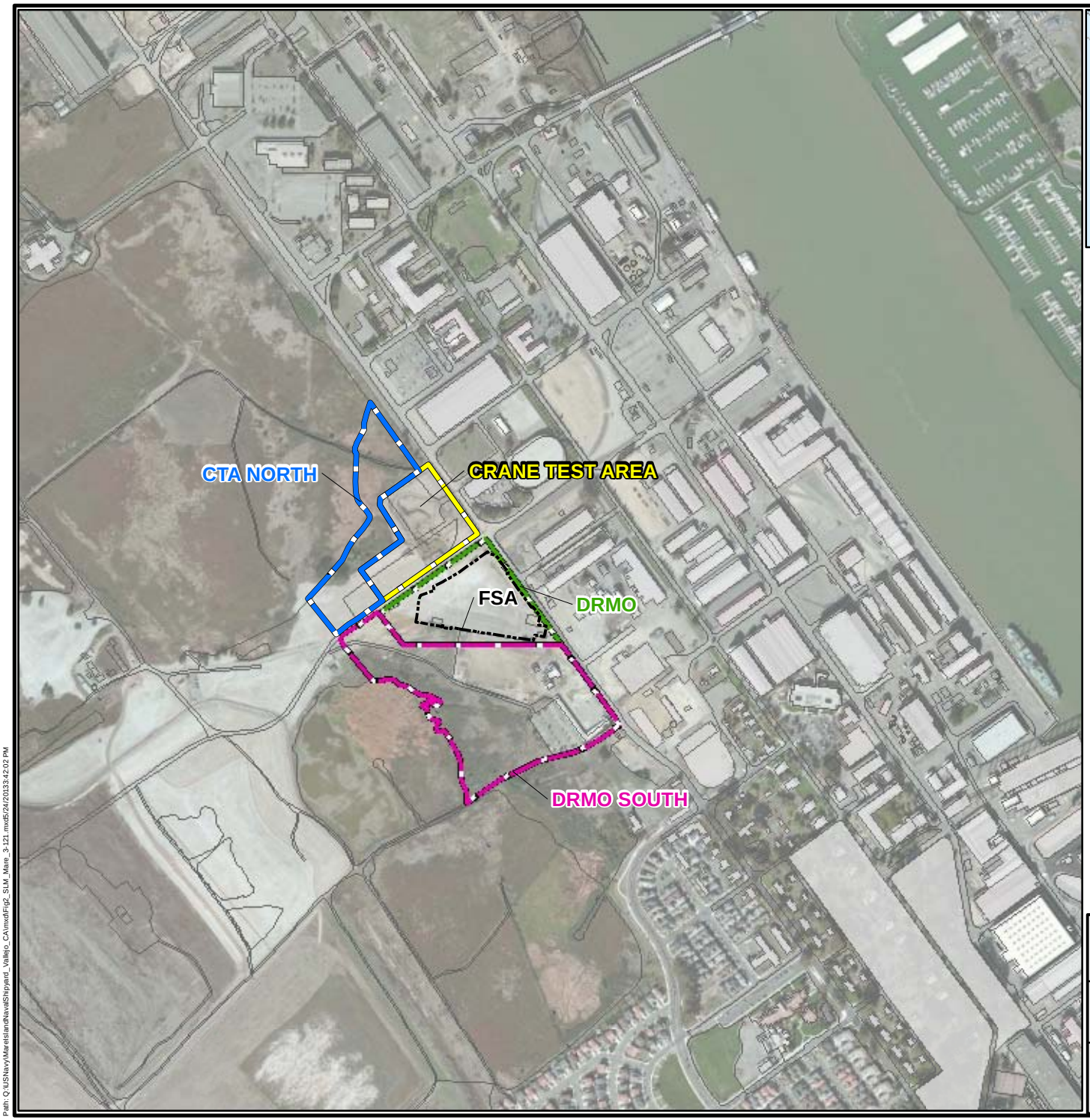

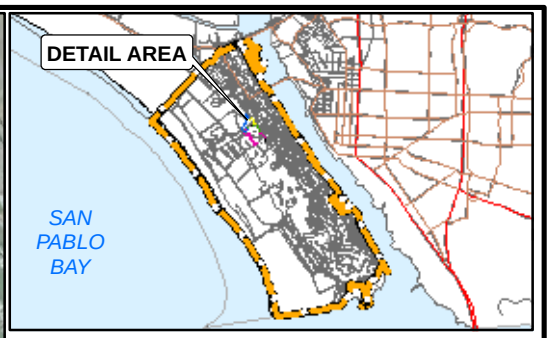

LEGEND:

- DRMO BOUNDARY
- DRMO SOUTH SITE BOUNDARY
- CTA NORTH SITE BOUNDARY
- CTA BOUNDARY (LENNAR MARE ISLAND)
- NAVY HISTORICAL BUILDING

ACRONYMS:
CTA - CRANE TEST AREA
DRMO - DEFENSE REUTILIZATION AND MARKETING OFFICE
FSA - FENCED SCRAPYARD AREA

SOURCE:
ENVIRONMENTAL SYSTEMS RESEARCH INSTITUTE (ESRI) USA SPATIAL FEATURE CLASSES. NAD83 STATE PLANE COORDINATE SYSTEM ZONE VI (FEET)
BASE MAP WAS OBTAINED FROM TREVET INC.
IMAGE © 2010 MICROSOFT CORPORATION AND ITS DATA SUPPLIERS, ACCESSED VIA ESRI ONLINE ON 3/26/13.

0 800 1,600
SCALE IN FEET

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

SITE LOCATION MAP

ARCADIS

FIGURE 2

CITY: SAN FRANCISCO DIV/GROUP: ENV/GIS DB: User: MEsifanos LD: PIC: PM: TM:
PROJECT: FE0401 0000
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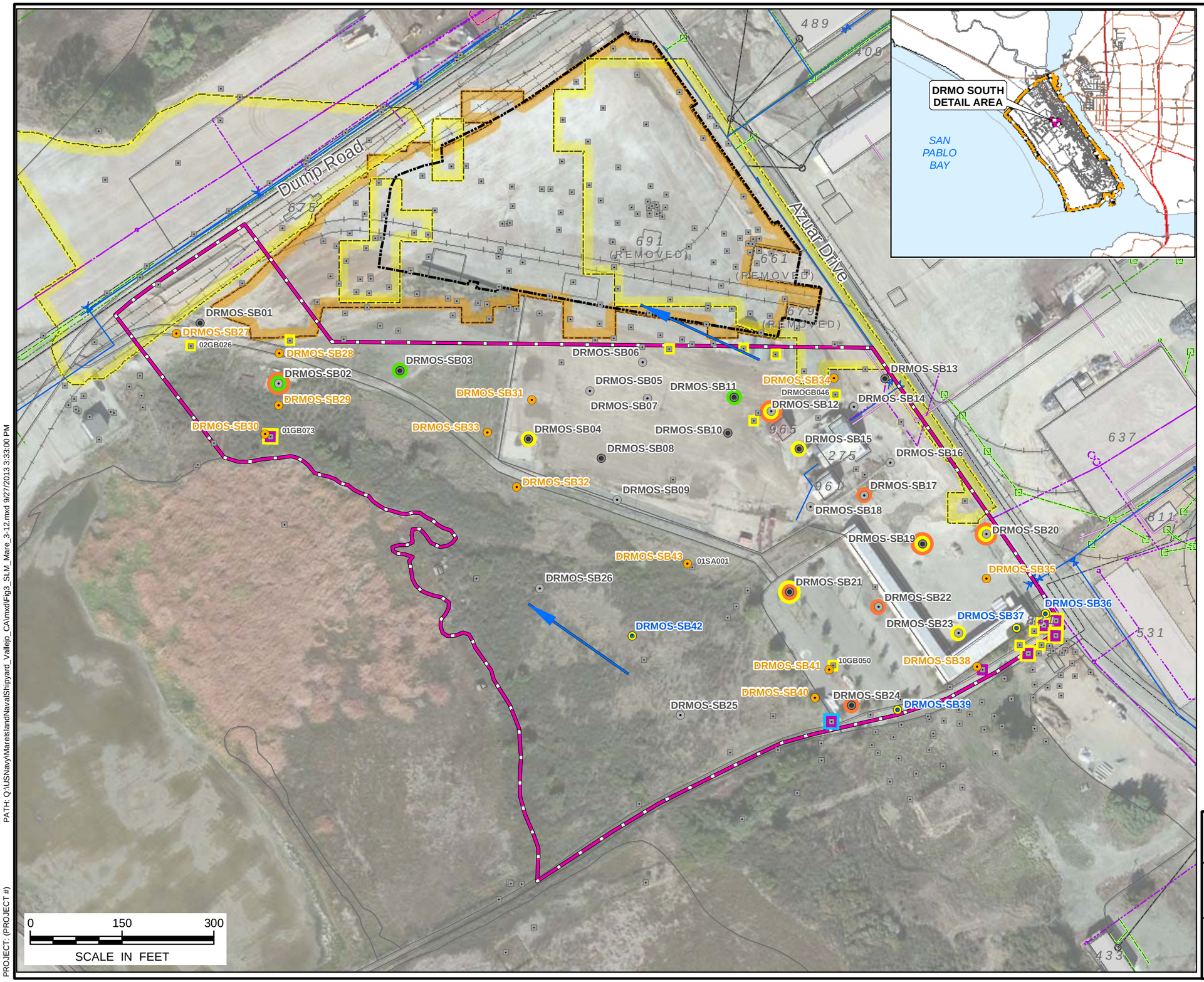

LEGEND

- SI SOIL SAMPLE LOCATION (2012)
- SI SOIL AND GROUNDWATER SAMPLE LOCATION (2012)
- PREVIOUSLY COLLECTED SAMPLE LOCATIONS
- PROPOSED SOIL/GROUNDWATER SAMPLING LOCATION
- PROPOSED SOIL SAMPLING LOCATION
- GROUNDWATER FLOW DIRECTION
- FENCED SCRAPYARD AREA
- DRMO SOUTH SITE BOUNDARY
- PCA EXCAVATION FOOTPRINT
- DRMO NTCRA BOUNDARY
- NAVY HISTORICAL BUILDING
- ELECTRICAL LINES
- SALTWATER PIPELINES
- GAS PIPELINES
- SANITARY SEWER PIPELINES
- WATER PIPELINES
- STORM DRAIN LINES
- INDUSTRIAL WASTE PIPELINES
- RAILROADS
- ARSENIC CONCENTRATION ABOVE SCREENING CRITERIA
- B(a)P CONCENTRATION ABOVE SCREENING CRITERIA
- DIESEL/MOTOR OIL CONCENTRATION ABOVE SCREENING CRITERIA
- LEAD CONCENTRATION ABOVE SCREENING CRITERIA
- AROCLOR 1260 CONCENTRATION ABOVE SCREENING CRITERIA

ACRONYMS:
B(a)P - BENZO(a)PYRENE
DRMO - DEFENSE REUTILIZATION AND MARKETING OFFICE
NTCRA - NON-TIME CRITICAL REMOVAL ACTION
PCA - PETROLEUM CORRECTIVE ACTION
RI - REMEDIAL INVESTIGATION
SI - SITE INSPECTION

SOURCE:
GOOGLE AERIAL IMAGE, DATE 8/4/2002
BASE MAP WAS OBTAINED FROM TREVET INC.

SOIL SCREENING CRITERIA ARE:
36 MILLIGRAMS PER KILOGRAM (mg/kg) FOR ARSENIC
0.21 mg/kg FOR B(a)P
100 mg/kg FOR DIESEL
500 mg/kg FOR MOTOR OIL
320 mg/kg FOR LEAD
1 mg/kg FOR AROCLOR 1260

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

DRMO SOUTH PROPOSED RI SAMPLING LOCATIONS

ARCADIS

FIGURE
3

CITY: SF DIV/GROUP: ENV/IM DB: MESTIFANOS LD: PIC: PM: TM: TR:
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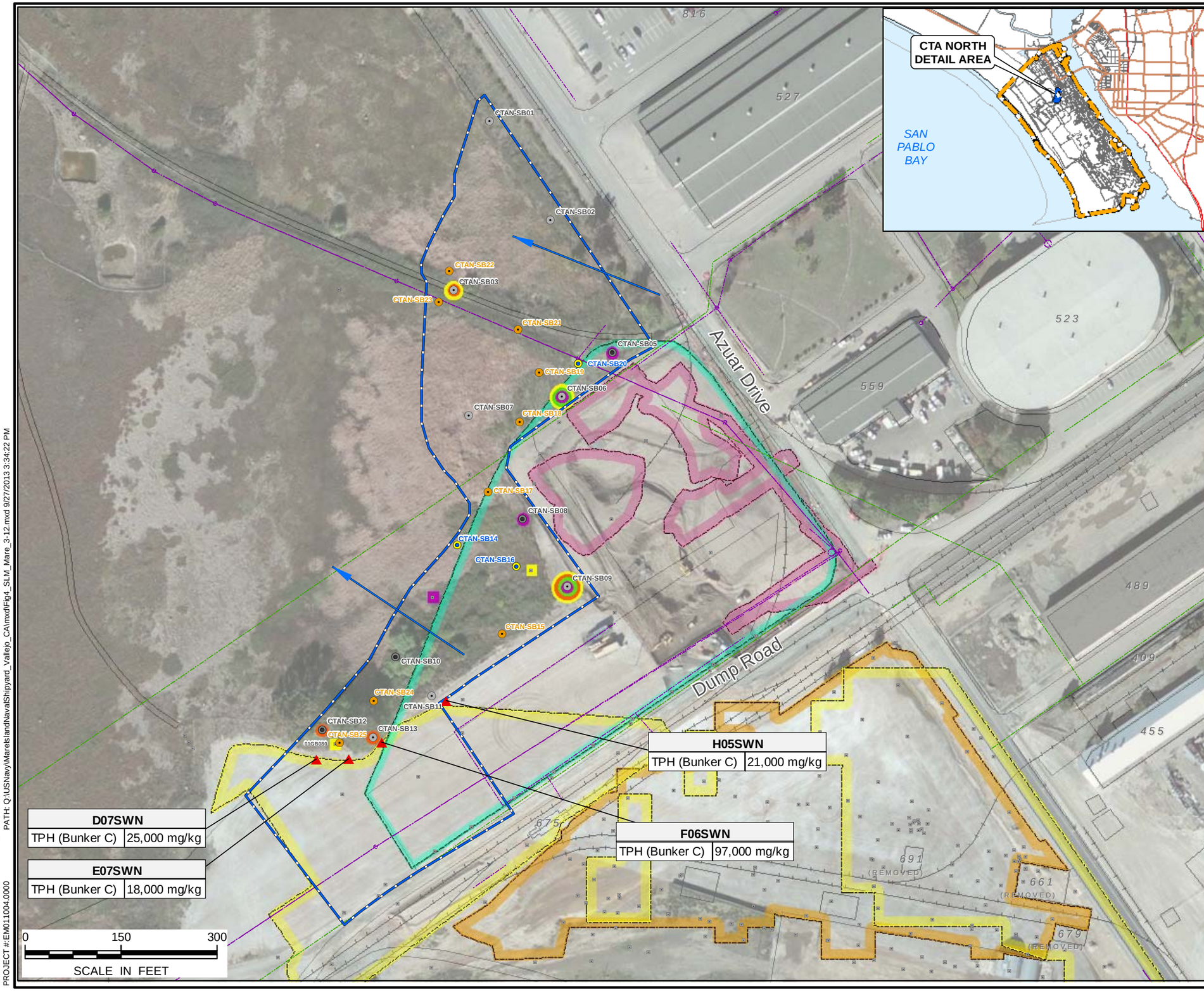


LEGEND

- SI SOIL SAMPLE LOCATION (2012)
- SI SOIL AND GROUNDWATER SAMPLE LOCATION (2012)
- PREVIOUSLY COLLECTED SAMPLE LOCATIONS
- SIDEWALL SAMPLE, CONCENTRATION ABOVE TPH CRITERION (PCA)
- GROUNDWATER FLOW DIRECTION
- ELECTRICAL UTILITY LINE
- SALTWATER UTILITY LINE
- CTA NORTH SITE BOUNDARY
- PCA EXCAVATION FOOTPRINT
- FILL AREA 1925-1942
- DRMO NTCRA BOUNDARY
- LMI CTA REMOVAL AREA FOOTPRINT
- NAVY HISTORICAL BUILDING
- RAILROADS
- PROPOSED SOIL/GROUNDWATER SAMPLING LOCATION
- PROPOSED SOIL SAMPLING LOCATION
- ARSENIC CONCENTRATION ABOVE SCREENING CRITERIA
- B(a)P CONCENTRATION ABOVE SCREENING CRITERIA
- LEAD CONCENTRATION ABOVE SCREENING CRITERIA
- DIESEL/MOTOR OIL CONCENTRATION ABOVE SCREENING CRITERIA

ACRONYMS:
B(a)P - BENZO(a)PYRENE
CTA - CRANE TEST AREA
LMI - LENNAR MARE ISLAND
NTCRA - NON-TIME CRITICAL REMOVAL ACTION
PCA - PETROLEUM CORRECTIVE ACTION
RI - REMEDIAL INVESTIGATION
SI - SITE INSPECTION
TPH - TOTAL PETROLEUM HYDROCARBONS

SOURCE:
GOOGLE AERIAL IMAGE, DATE 8/4/2002
BASE MAP WAS OBTAINED FROM TREVET INC.

SOIL SCREENING CRITERIA ARE:
36 MILLIGRAMS PER KILOGRAM (mg/kg) FOR ARSENIC
0.21 mg/kg FOR B(a)P
320 mg/kg FOR LEAD
100 mg/kg FOR DIESEL
500 mg/kg FOR MOTOR OIL

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

CTA NORTH PROPOSED RI SAMPLING LOCATIONS

ARCADIS

FIGURE
4

CITY: SF DIV/GROUP: ENV/IM DB: MESTIFANOS LD: PIC: PM: TM: TR:
PROJECT #:EM011004.0000 PATH: Q:\USNavy\MareIslandNavalShipyard_Vallejo_CA\mxd\Fig4_SLM_Mare_3-12.mxd 9/27/2013 3:34:22 PM

SOURCES/ RELEASE MECHANISMS	MIGRATION PATHWAYS	EXPOSURE MEDIA	EXPOSURE ROUTE	Potential Receptors				
				On-site Commercial/ Industrial Workers	On-site Utility and Construction Workers	Recreational Users	Residents (Hypothetical)	Ecological
Historical Releases to Soil or Groundwater from Former Activities	Volatilization	Air Vapors	Inhalation (Indoor Air)	--	--	--	--	--
			Inhalation (Outdoor Air)	○	○	○	○	--
	Leaching	Groundwater	Ingestion	--	--	--	--	○
			Dermal Contact	--	○	--	--	--
			Inhalation (from Potable Water)	--	--	--	--	--
	Wind Erosion and Transport	Dust Particles	Inhalation (Outdoor Air)	○	○	○	○	○
	Surface-Water Runoff Erosion and Transport	Surface and/or Subsurface Soil	Ingestion	○	○	○	○	○
			Dermal Contact	○	○	○	○	○
	Plant/Animal Uptake	Plant/Animal Tissue	Ingestion	--	--	--	--	○

○ Exposure pathway is complete or potentially complete;

-- Incomplete exposure pathway.

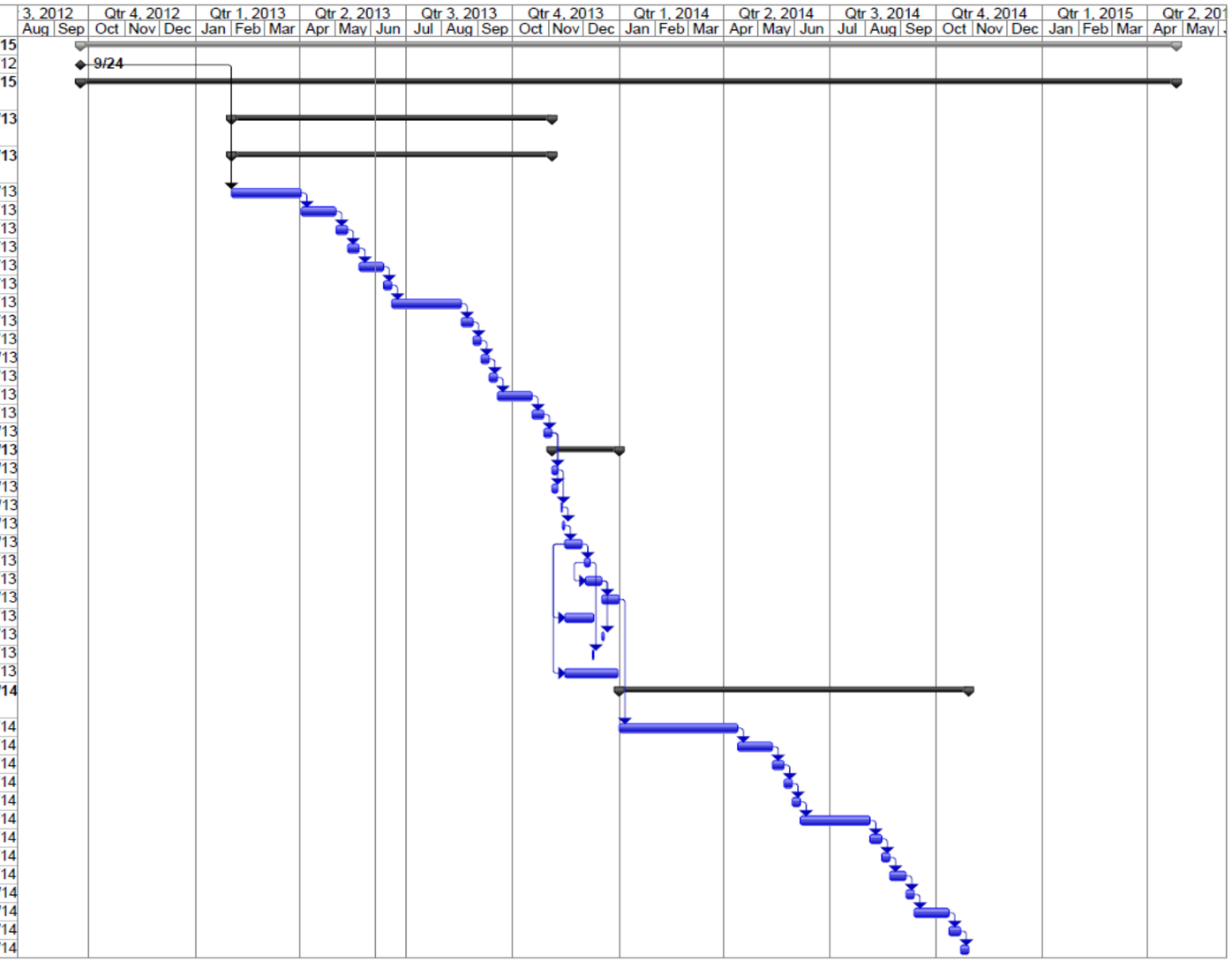
MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

EXPOSURE PATHWAY SUMMARY

ARCADIS

FIGURE
5

ID	Task Name	Duration	Start	Finish
0	**Mare Island - Remedial Investigation**	**655 days**	**Mon 9/24/12**	**Sun 4/26/15**
1	Contract Award	0 edays	Mon 9/24/12	Mon 9/24/12
2	**Work Element No. 1 - Project Management, Meetings, Administrative Support**	**655 days**	**Mon 9/24/12**	**Sun 4/26/15**
5	**Work Element No. 2 - Remedial Investigation (RI) Work Plan**	**191 days**	**Fri 2/1/13**	**Mon 11/4/13**
6	**Remedial Action Work Plan (including all supporting plans)**	**191 days**	**Fri 2/1/13**	**Mon 11/4/13**
7	Internal Draft RI Work Plan (WP/SSHP/SAP)	60 edays	Fri 2/1/13	Tue 4/2/13
8	Navy Review	30 edays	Tue 4/2/13	Thu 5/2/13
9	Response to Navy RPM/ROICC Comments	10 edays	Thu 5/2/13	Sun 5/12/13
10	Revised Internal Draft SAP QAO Comments	10 edays	Sun 5/12/13	Wed 5/22/13
11	Navy/QAO Review and Comment Resolution	21 edays	Wed 5/22/13	Wed 6/12/13
12	Draft RI Work Plan (WP/SSHP/SAP)	7 edays	Wed 6/12/13	Wed 6/19/13
13	Regulatory Review	60 edays	Wed 6/19/13	Sun 8/18/13
14	Response to Regulatory Comments	10 edays	Sun 8/18/13	Wed 8/28/13
15	Internal Draft Final RI Work Plan (WP/SSHP/SAP)	7 edays	Wed 8/28/13	Wed 9/4/13
16	Navy/QAO Review and Approval	7 edays	Wed 9/4/13	Wed 9/11/13
17	Draft Final RI Work Plan (WP/SSHP/SAP)	7 edays	Wed 9/11/13	Wed 9/18/13
18	Regulatory Review	30 edays	Wed 9/18/13	Fri 10/18/13
19	Response to Regulatory Comments	10 edays	Fri 10/18/13	Mon 10/28/13
20	Final RI Work Plan (WP/SSHP/SAP)	7 edays	Mon 10/28/13	Mon 11/4/13
21	**Work Element No. 3 - Field Work**	**39 days**	**Mon 11/4/13**	**Tue 12/31/13**
22	Pre-field Activities	5 days	Mon 11/4/13	Fri 11/8/13
23	Permitting	5 days	Mon 11/4/13	Fri 11/8/13
24	Mobilization and Site Preparation	1 day	Tue 11/12/13	Tue 11/12/13
25	Utility Clearance	2 days	Wed 11/13/13	Thu 11/14/13
26	Soil Sampling and Well Installation	10 days	Fri 11/15/13	Fri 11/29/13
27	Groundwater Sampling	5 days	Mon 12/2/13	Fri 12/6/13
28	Laboratory Analysis	10 days	Tue 12/3/13	Mon 12/16/13
29	Data Validation	10 days	Tue 12/17/13	Tue 12/31/13
30	Biological Monitoring and Avoidance	16 days	Fri 11/15/13	Mon 12/9/13
31	Land Survey	2 days	Tue 12/17/13	Wed 12/18/13
32	Site Restoration and Demobilization	1 day	Mon 12/9/13	Mon 12/9/13
33	IDW Management, Profiling and Disposal	30 days	Fri 11/15/13	Mon 12/30/13
34	**Work Elements No. 4 - Remedial Investigation Reports**	**212 days**	**Tue 12/31/13**	**Tue 10/28/14**
35	Internal Draft RI Reports	102 edays	Tue 12/31/13	Sat 4/12/14
36	Navy Review	30 edays	Sat 4/12/14	Mon 5/12/14
37	Response to Navy Comments	10 edays	Mon 5/12/14	Thu 5/22/14
38	Navy QAO SAP Review	7 edays	Thu 5/22/14	Thu 5/29/14
39	Draft RI Report	7 edays	Thu 5/29/14	Thu 6/5/14
40	Regulatory Review	60 edays	Thu 6/5/14	Mon 8/4/14
41	Response to Regulatory Comments	10 edays	Mon 8/4/14	Thu 8/14/14
42	Internal Draft Final RI Report	7 edays	Thu 8/14/14	Thu 8/21/14
43	Navy Review and Approval	14 edays	Thu 8/21/14	Thu 9/4/14
44	Draft Final RI Report	7 edays	Thu 9/4/14	Thu 9/11/14
45	Regulatory Review	30 edays	Thu 9/11/14	Sat 10/11/14
46	Response to Regulatory Comments	10 edays	Sat 10/11/14	Tue 10/21/14
47	Final RI Report	7 edays	Tue 10/21/14	Tue 10/28/14



e days = calendar days
days = working days

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

PROJECT SCHEDULE

ARCADIS

FIGURE
6

Appendix A: Sampling and Analysis Plan

SAP Worksheet #1 — Title and Approval Page

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

November 2013

REMEDIAL INVESTIGATION FOR
DEFENSE REUTILIZATION AND MARKETING OFFICE SOUTH
AND CRANE TEST AREA NORTH
FORMER MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

Prepared for:
Department of the Navy
Base Realignment and Closure
Program Management Office West
1455 Frazee Road
San Diego, California

Prepared by:
ARCADIS U.S., Inc.
2000 Powell Street, 7th Floor
Emeryville, CA 94608

Prepared under:
Contract N62473-11-D-2226
Task Order 0004
DCN: AUS-2226-0004-0001

Review Signature: _____ Date 11/5/2013
Lyndi Mott
Quality Assurance Manager, ARCADIS U.S., Inc.

Approval Signature: _____ Date November 22, 2013
Joseph Michalowski
Quality Assurance Officer, NAVFAC SW

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EXECUTIVE SUMMARY

On behalf of the United States Department of the Navy (Navy), ARCADIS U.S., Inc. (ARCADIS) has prepared this Sampling and Analysis Plan (SAP, Appendix A of the Remedial Investigation Work Plan) to conduct a Remedial Investigation (RI) for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North, located at the former Mare Island Naval Shipyard (MINS) in Vallejo, California. In general, the objectives of the RI are to characterize site conditions, evaluate the nature and extent of affected soil and groundwater, refine the conceptual site model (CSM), and assess potential risk to human health and the environment. Information obtained during this process will guide future decisions made regarding DRMO South and CTA North. This SAP provides guidance on sampling, analysis, and quality assurance (QA) to acquire the data necessary to meet the RI objectives.

DRMO South and CTA North are located within the north-central portion of MINS. DRMO South occupies land to the south of Dump Road and the DRMO site and west of Azuar Drive, while CTA North occupies the upland area to the north of Dump Road and the CTA site and west of Azuar Drive. The CTA was transferred to the City of Vallejo, which then transferred the CTA to Mare Island redeveloper, Lennar Mare Island, in 2001. Site inspections (SIs) were conducted at DRMO South and CTA North in October and November 2012 in response to a California Department of Toxic Substances Control (DTSC) request for additional information and/or data to address the possibility that affected soil and groundwater similar to that observed at the CTA may also be present on the adjacent Navy property (DTSC 2009). The Navy also identified DRMO South, located in the vicinity of CTA North, for further investigation based on the findings of the Internal Draft Investigation Area (IA) H2 RI Report (Tetra Tech EM, Inc. 2000). IA H2 included DRMO South as well as portions of CTA North. Results of the 2012 SIs demonstrated that chemicals of potential concern (COPCs), including metals, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), total petroleum hydrocarbons as diesel (TPH-d), and total petroleum hydrocarbons as motor oil (TPH-mo), were present in soil at concentrations exceeding human health and/or ecological screening criteria and warranted additional characterization. COPCs detected in groundwater at concentrations exceeding human health and/or ecological screening criteria included metals, pesticides, PAHs, and volatile organic compounds.

The goals of the RI are to gather sufficient data to: (1) further evaluate and/or delineate COPCs identified in soil and groundwater at DRMO South and CTA North; (2) determine whether COPCs identified in soil and groundwater at the sites would result in unacceptable health risks to future receptors; and (3) update the CSM. Data obtained during the RI field work will supplement historical data gathered during previous field efforts.

The following scope of work has been defined to achieve the RI goals:

- Collect 48 soil samples from 17 boring locations at DRMO South. Soil samples will be analyzed for metals, PAHs, PCBs, and/or total petroleum hydrocarbons-extractable (TPH-e).
- Collect four groundwater samples from four temporary monitoring well locations at DRMO South. Groudnwater samples will be analyzed for metals, PAHs, PCBs, and/or TPH-e.

- Collect 39 soil samples from 12 boring locations at CTA North. Soil samples will be analyzed for PAHs, PCBs, TPH-e, and/or metals.
- Collection of three groundwater samples from three temporary monitoring well locations at CTA North. Groundwater samples will be analyzed for PAHs, PCBs, TPH-e, and/or metals.

Soil samples will be collected from two or more depth intervals at each boring. Soil samples will be collected from depths of up to 15 feet below ground surface (bgs) at DRMO South and up to 10 feet bgs at CTA North based on the vertical extent of impacts observed during previous investigations. The depths of the deeper samples (i.e., samples collected from below 0.5 feet bgs) may be biased if visual observations and/or photo ionization detector screening of the soil column indicate the possible presence of COPCs. Groundwater samples will be collected based on the observed depth of the water table and the final screening interval. In general, groundwater samples will be collected from the uppermost water-bearing zone.

ARCADIS will prepare separate RI Reports for CTA North and DRMO South to document the results of the RI field and data analysis activities, present the results of the baseline human health and ecological risk assessments, and provide recommendations for further action, as appropriate. The RI Reports will be certified by a California-licensed professional engineer or geologist who is actively involved in the project.

This SAP will be used as a reference document by all field and laboratory personnel engaged in sampling and analysis for the project. The document will be provided to individuals listed in Worksheet #3. Included in this SAP are data quality objectives, field sampling procedures, QA/quality control requirements, and data gathering methods that will be used during the project. This SAP was prepared in accordance with the requirements of the Uniform Federal Policy for Quality Assurance Plans (USEPA 2005) and United States Environmental Protection Agency (USEPA) Guidance for Quality Assurance Project Plans, EPA QA/G-5 (USEPA 2002a).

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Figure 1: Regional Map
Figure 2: Site Location Map
Figure 3: DRMO South Proposed RI Sampling Locations
Figure 4: CTA North Proposed RI Sampling Locations
Figure 5: Exposure Pathways

Attachments

Attachment 1: Field Forms
Attachment 2: Laboratory Certification
Attachment 3: Laboratory Standard Operating Procedures
Attachment 4: Control Limits

ACRONYMS AND ABBREVIATIONS

±	plus or minus
>	greater than
≤	less than or equal to
%	percent
ºC	degrees Celsius
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
AES	atomic emission spectroscopy
APP	Accident Prevention Plan
ARCADIS	ARCADIS U.S., Inc.
AWQC	Ambient Water Quality Criteria
BCT	BRAC Cleanup Team
B(a)P	benzo(a)pyrene
bgs	below ground surface
BRAC	Base Realignment and Closure
CA	corrective action
CAS	Chemical Abstract Service
CCC	calibration check compound
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHHSL	California Human Health Screening Level
CLP NFG	Contract Laboratory Program National Functional Guidelines
COC	chain of custody
COPC	chemical of potential concern
CSM	conceptual site model
CSO	Caretaker Site Office
CTA	Crane Test Area
CVAA	cold vapor atomic absorption
DCN	Document Control Number
DL	Detection Limit
DoD	Department of Defense
DQA	data quality assessment
DQI	data quality indicator
DQO	data quality objectives
DRMO	Defense Reutilization and Marketing Office
DTSC	Department of Toxic Substances Control
EcoSSL	Ecological Soil Screening Level
ELAP	Environmental Laboratory Accreditation Program
EMAX	EMAX Laboratories, Inc.
EPC	exposure point concentration
ESL	Environmental Screening Level

FSA	Fenced Scrapyard Area
GC/MS	Gas chromatograph mass spectrometer
H&S	Health and Safety
IA	Investigation Area
ICAL	initial calibration
ICP	inductively coupled plasma
ICS	interference check solution
ICV	second source calibration verification
IDW	investigation-derived waste
IR10	Installation Restoration Site 10
IR14	Installation Restoration Site 14
IW	industrial wastewater
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDC	Laboratory Data Consultants
LOD	limit of detection
LOQ	limit of quantitation
MCL	maximum contaminant level
MDL	maximum detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MINS	Mare Island Naval Shipyard
MS	matrix spike
MS/MSD	matrix spike/matrix spike duplicate
NAVFAC SW	Naval Facilities Engineering Command Southwest
Navy	United States Department of the Navy
NIRIS	Naval Installation Restoration Information Solution
NTCRA	non-time-critical removal action
ORNL	Oak Ridge National Laboratory
PA	preliminary assessment
PAH	polycyclic aromatic hydrocarbon
PAL	project action limit
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCA	Petroleum Corrective Action
PCB	polychlorinated biphenyl
PID	photo ionization detector
PM	project manager
PMO West	Program Management Office West
PQO	project quality objective
PT	proficiency testing
PVC	polyvinyl chloride
QA	quality assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer

QC	quality control
QCM	Quality Control Manager
QL	quantitation limit
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RF	response factor
RI	remedial investigation
ROICC	Resident Officer In Charge of Construction
RPD	relative percent difference
RPM	Remedial Project Manager
RSL	Regional Screening Level
Water Board	California Regional Water Quality Control Board, San Francisco Bay Region
SAP	Sampling and Analysis Plan
SI	site inspection
SIM	selected ion monitoring
SL	screening level
SOP	standard operating procedure
SPCC	system performance check compound
SSHP	Site Safety and Health Plan
SVOC	semivolatile organic compound
SWDIV	Southwest Division Naval Facilities Engineering Command
SWMU	solid waste management unit
TDS	total dissolved solids
TPH	total petroleum hydrocarbons
TPH-e	total petroleum hydrocarbons - extractable
TPH-p	total petroleum hydrocarbons - purgeable
TPH-d	total petroleum hydrocarbons - diesel
TPH-mo	total petroleum hydrocarbons - motor oil
TtEM	Tetra Tech EM, Inc.
UCL	upper confidence limit
UFP	Uniform Federal Policy
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USEPA	United States Environmental Protection Agency
VOA	volatile organic analysis
VOC	volatile organic compound
Weston	Weston Solutions, Inc.

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SAP Worksheet #2 — SAP Identifying Information

Site Name/Number: Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North
Operable Unit: Not Applicable
Contractor Name: ARCADIS U.S., Inc.
Contract Number: N62473-11-D-2226
Contract Title: Performance-Based Environmental Multiple Award Contract (PERMAC) II, Remedial Investigation for Crane Test Area North and Defense Reutilization and Marketing Office South at Former Mare Island Naval Shipyard, Vallejo, California
Work Assignment Number (optional): TO 0004

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)Manual (USEPA 2005) and Guidance for Quality Assurance Project Plans, QA/G-5 (USEPA 2002).

2. Identify regulatory program: CERCLA (Comprehensive Environmental Response, Compensation, and Liability Act)

3. This is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Scoping Session	03 October 2012

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

Title	Date
Final SAP for the Site Investigation for Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California	September 2012

6. List organizational partners (stakeholders) and connection with lead organization:
   - United States Environmental Protection Agency (USEPA)
   - California Department of Toxic Substances Control (DTSC).
   - Regional Water Quality Control Board, San Francisco Region (Water Board)

7. Lead organization:
   United State Department of the Navy

8. 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:
   Not applicable. No SAP elements have been omitted from this SAP.

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 Procedures (SOP) Requirements Table Sample Location Maps	
19	Analytical Methods/SOP Requirements Table	
20	Field Quality Control Sample Summary Table	
21	Project Sampling SOP References Table Sampling SOPs	

SAP Worksheet #2 – continued

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
Sampling Tasks (continued)		
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 Table	
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 (COC) Form and Seal	
Quality Control Samples		
28	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	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	

Notes:
COC Chain of Custody
QA/QC Quality Assurance/Quality Control
SOP Standard Operating Procedure
UPF QAPP Uniform Federal Policy for Quality Assurance Plan

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SAP Worksheet #3 — Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number	Mailing Address	E-mail Address
Christopher Dirscherl	Remedial Project Manager (RPM)	Base Realignment and Closure (BRAC) Program Management Office West (PMO West)	(619) 532-0785	1455 Frazee Road, Suite 900 San Diego, CA 92108-4310	christopher.dirscher @navy.mil
Heather Wochnick	Lead RPM	BRAC PMO West	(619) 532-0763	1455 Frazee Road, Suite 900 San Diego, CA 92108-4310	heather.wochnick@navy.mil
Janet Lear	BRAC Environmental Coordinator	BRAC PMO West	(619) 532-0976	1455 Frazee Road, Suite 900 San Diego, CA 92108-4310	janet.lear@navy.mil
Joseph Michalowski	Quality Assurance Officer (QAO)	Naval Facilities Engineering Command Southwest (NAVFAC SW)	(619) 532-4125	1220 Pacific Hwy San Diego, CA 92132	joseph.michalowski @navy.mil
Melecio Asuncion	Caretaker Site Office Representative	NAVFAC SW	(510) 772-7639	410 Palm Ave, Bldg. 1, Ste. 161 San Francisco, CA 94130	melecio.asuncion@navy.mil
Izzat Amadea	Resident Officer in Charge of Construction	NAVFAC SW	(510) 749-5945	2450 Saratoga Street, Suite 200 Alameda, CA 94501	izzat.amadea@navy.mil
Diane Silva	Administrative Records Manager	NAVFAC SW	(619) 532-3676	1220 Pacific Hwy Bldg. 1, 3rd Floor San Diego, CA 92132	diane.silva@navy.mil
Janet Naito	Project Manager (PM)	DTSC	(510) 540-3833	700 Heinz Avenue, Suite 200 Berkeley, CA 94710-2721	janet.naito@dtsc.ca.gov
Elizabeth Wells	Regulatory Agency Representative	Water Board	(510) 622-2440	1515 Clay Street, #1400 Oakland CA 94612	elizabeth.wells@ waterboards.ca.gov

SAP Worksheet #3 – continued

Name of SAP Recipients	Title/Role	Organization	Telephone Number	Mailing Address	E-mail Address
Carolyn d'Almeida	Regulatory Agency Representative	USEPA	(415) 972-3150	75 Hawthorne Street Mail Code SFD-8-1 San Francisco, CA 94105	dalmeida.carolyn @epa.gov
Hoa Voscott	PM	ARCADIS	(510) 596-9609	2000 Powell Street, 7th Floor Emeryville, CA 94608	hoa.voscott@arcadis-us.com
Amy Goldberg Day	Project Toxicologist	ARCADIS	(510) 596-9507	2000 Powell Street, 7th Floor Emeryville, CA 94608	amy.goldbergday@arcadis-us.com
Dennis Dyke	Project Chemist	ARCADIS	(510) 596-9669	2000 Powell Street, 7th Floor Emeryville, CA 94608	dennis.dyke@arcadis-us.com
Emily Sheu	Project Engineer & Field Team Lead	ARCADIS	(510) 596-9653	2000 Powell Street, 7th Floor Emeryville, CA 94608	emily.sheu@arcadis-us.com
Lyndi Mott	Project Quality Assurance Manager	ARCADIS	(713) 953-4829	2929 Briarpark Drive, Suite 300 Houston, TX, 77042	lyndi.mott@arcadis-us.com
Will Rice	PM (analytical services)	Curtis & Tompkins Laboratories	(510) 204-2221	2323 5th Street Berkeley, CA 94710	Will.rice@ctberk.com
Pei Geng	PM (data validation)	Laboratory Data Consultants	(760) 634-0437	7750 El Camino Real, Suite 2L Carlsbad, CA. 92009	pgeng@lab-data.com

Notes:

BRAC	Base Realignment and Closure
DTSC	Department of Toxic Substances Control
NAVFAC SW	Naval Facilities Engineering Command Southwest
PM	Project Manager
PMO West	Program Management Office West
Water Board	Regional Water Quality Control Board
SAP	Sampling and Analyses Plan

SAP Worksheet #4 — Project Personnel Sign-Off Sheet

Name	Organization/Title/Role	Telephone Number (optional)	Signature/e-mail receipt	SAP Section Reviewed	Date SAP Read
Hoa Voscott	ARCADIS / Project Manager	(510) 596-9609		All	
Lyndi Mott	ARCADIS / Project QA Manager	(713) 953-4829		All	
Amy Goldberg Day	ARCADIS / Project Toxicologist	(510) 596-9507		All	
Dennis Dyke	ARCADIS / Project Chemist	(510) 596-9669		All	
Emily Sheu	ARCADIS / Project Engineer & Field Team Lead	(510) 596-9653		All	
Will Rice	Curtis & Tompkins Laboratories / Project Manager	(510) 204-2221		Worksheets 12, 15, 19, 20, 23, 24, 25, 28, and 30	
Pei Geng	Laboratory Data Consultants, Inc.	(760) 634-0437		Worksheets 12, 14, 15, 19, 20, 23, 24, 25, 28, 30 to 37, plus Laboratory SOPs and Control Limits in Attachments 3 and 4	

Notes:
QA Quality Assurance
SAP Sampling and Analyses Plan

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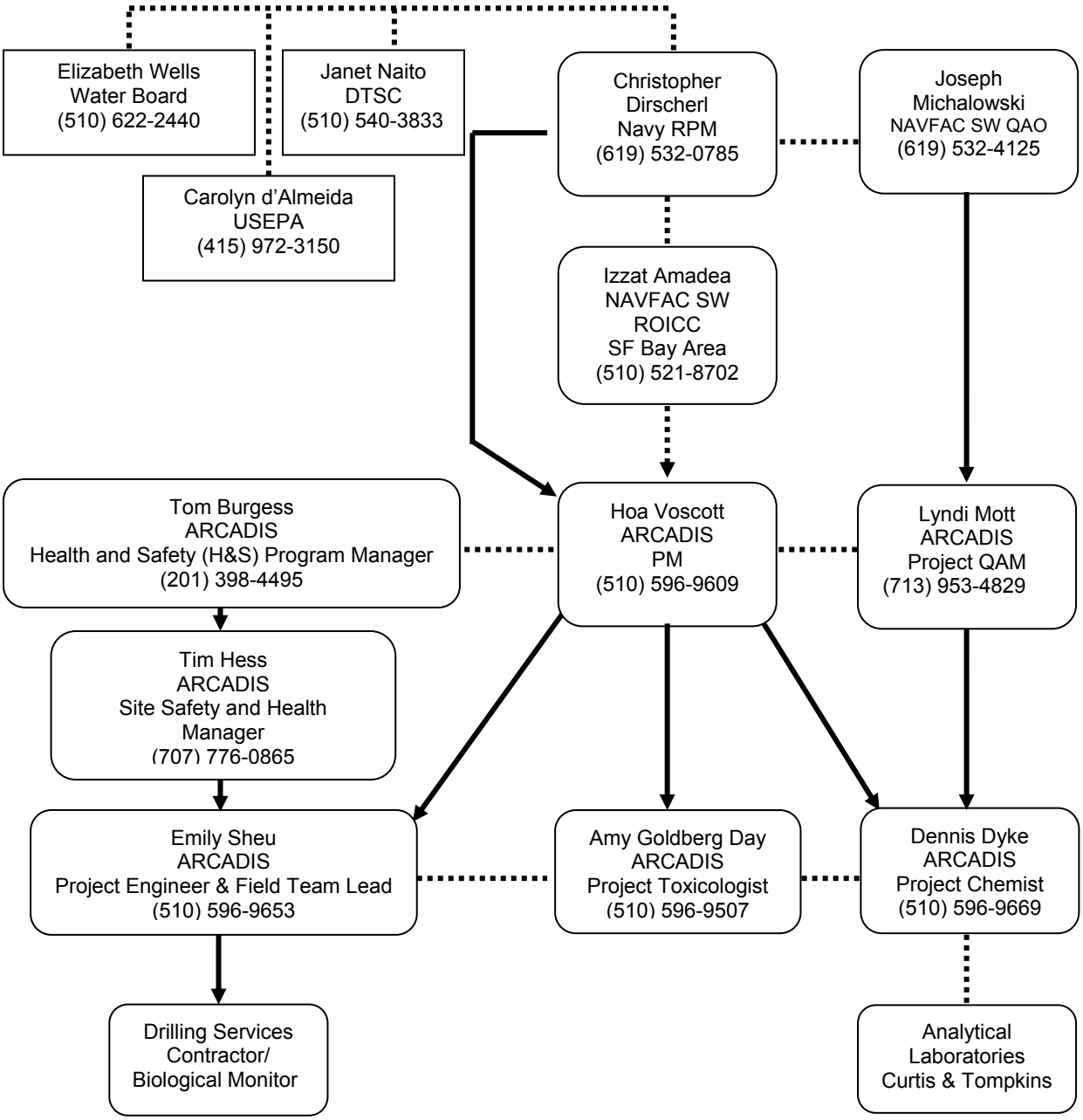
SAP Worksheet #5 — Project Organizational Chart

Lines of Authority ——— Lines of Communication



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SAP Worksheet #6 — Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Notification – Field sampling activities	ARCADIS PM	Hoa Voscott	(510) 596-9609	One week prior to mobilization of field sampling teams an e-mail or phone call to Navy RPM from the ARCADIS PM to confirm the sampling start date and duration.
Notification – Field sampling activities	Navy RPM	Chris Dirscherl	(619) 532-0785	Three days prior to start of field activities the Navy RPM will notify the regulatory agencies.
Notification – Laboratory notification and coordination	ARCADIS Project Chemist or Project Engineer	Dennis Dyke or Emily Sheu	(510) 596-9669 (510) 596-9653	One week prior to mobilization of field sampling team, phone call to EMAX Lab from Trevet Project Chemist to coordinate sample supplies and courier pickup of samples.
Stop work	NAVFAC SW QAO	Joseph Michalowski	(619) 532-4125	If a quality issue arises that warrants a stop work order, an immediate phone call to the ARCADIS QAM from the NAVFAC S QAO will occur and a stop work order will be issued.
Stop work	Navy RPM	Chris Dirscherl	(619) 532-0785	If any issue arises that warrants a stop work order, an immediate phone call to the ARCADIS PM from the Navy RPM will occur and a stop work order will be issued.
Stop work	NAVFAC SW Safety Officer	Almario Erasquin	(619) 556-7938	If a safety issue arises that warrants a stop work order, an immediate phone call to the ARCADIS Site Safety and Health Manager from the NAVFAC SW Safety Officer will occur and a stop work order will be issued.
Stop work	ARCADIS QAM	Lyndi Mott	(713) 953-4829	If a quality issue arises that warrants a stop work order, an immediate phone call to the NAVFAC SW QAO from the ARCADIS QAM will occur and a stop work order will be issued.
Stop work	ARCADIS Site Safety and Health Manager	Tim Hess	(707) 776-0865	If a safety issue arises that warrants a stop work determination, an immediate phone call to the ARCADIS Project Engineer/Field Team Lead from the ARCADIS Site Safety and Health Manager will occur and the ARCADIS Project Engineer/Field Team Lead will stop work.

SAP Worksheet #6 – continued

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Notification – Reporting laboratory data quality issues	Laboratory (Curtis & Tompkins) PM	Will Rice	(510) 204-2221	All QA/QC (quality assurance/quality control) issues will be reported by the Laboratory PM to the ARCADIS Project Chemist and Project Engineer in writing within two business days.
Notification – Field Change Request	ARCADIS Field Team Lead	Emily Sheu	(510) 596-9653	If a sampling issue arises during field activities that conflict with the approved SAP, an immediate phone call to the ARCADIS QAM from the ARCADIS Field Team Lead will be placed to discuss a field change request.
Notification – Field Change Request	ARCADIS QAM	Lyndi Mott	(713) 953-4829	If a field change request is warranted, then a written Field Change Request will be emailed from the ARCADIS QAM to the ARCADIS PM, Navy RPM, and NAVFAC SW QAO.
SAP Addendum	ARCADIS QAM	Lyndi Mott	(713) 953-4829	Any significant changes to the SAP will require the ARCADIS QAM (or designee) to prepare an addendum that will be approved by the NAVFAC SW QAO before any field activities begin.
Daily chain-of-custody (COC) reports and shipping documentation	ARCADIS Field Team Lead	Emily Sheu	(510) 596-9653	At the end of each field sampling day, the COC forms and shipping documentation (if applicable) will be submitted via fax or e-mail to the ARCADIS Project Chemist and ARCADIS Project Engineer.
Analytical corrective actions	ARCADIS Project Chemist	Dennis Dyke	(510) 596-9669	The ARCADIS Project Chemist will immediately notify the ARCADIS PM in writing of any analytical procedures that were not performed in accordance with this SAP. The ARCADIS Project Chemist will complete documentation of the non-conformance and corrective actions to be taken. The ARCADIS QAM (or designee) will be informed and will verify that the corrective actions have been implemented.
Notification - Release of analytical data	ARCADIS Project Chemist	Dennis Dyke	(510) 596-9669	The ARCADIS Project Chemist will review faxed/e-mailed data to verify that data quality is met as described in this SAP before the data are released. Analytical data will be released to the ARCADIS PM (or designee) after the ARCADIS Project Chemist has verified that the data are in accordance with the SAP requirements.

SAP Worksheet #6 – continued

Notes:

COC	Chain of Custody
NAVFAC SW	Naval Facilities Engineering Command Southwest
PM	Project Manager
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QA/QC	Quality Assurance/Quality Control
RPM	Regional Project Manager
Water Board	Regional Water Quality Control Board
SAP	Sampling and Analysis Plan

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SAP Worksheet #7 — Personnel Responsibilities and Qualifications Table

Name	Title/Role	Organizational Affiliation	Responsibilities
Joseph Michalowski	Quality Assurance Officer	NAVFAC SW	• Reviews and approves SAP • Provides Department of the Navy (Navy) oversight of ARCADIS Quality Assurance Program • Provides technical and administrative oversight of surveillance audit activities • Acts as point of contact for matters concerning quality assurance and the Navy Laboratory Quality Assurance Program • Coordinates training on matters pertaining to generation and maintenance of quality of data • Authorizes the suspension of project execution if quality assurance requirements are not adequately followed
Christopher Dirscherl	Navy RPM	BRAC PMO West	• Performs project management for the Navy • Ensures that the project scope of work requirements are fulfilled • Oversees the project cost and schedule • Provides formal technical direction to the ARCADIS project team, as needed • Acts as lead interface with agencies
Hoa Voscott	PM	ARCADIS	• Manages project activities and allocates resources and personnel to accomplish the project tasks and ensure that the project deliverables are completed on time and within budget • Acts as primary contact between the Navy RPM and Contractor • Supervises preparation of planning documents and sampling plans • Ensures adherence to the quality requirements of the contract, project scope of work, and plans • Ensures that all work is conducted in accordance with the Work Plan • Ensures that all work meets the requirements of the technical specifications and complies with applicable codes and regulations • Coordinates satisfactory resolution and completion of evaluation and acceptance report for nonconformance reports

SAP Worksheet #7 – continued

Name	Title/Role	Organizational Affiliation	Responsibilities
Lyndi Mott	QAM	ARCADIS	• Establishes and maintains the quality program • Documents that construction and chemical data acquisition activities are completed per the approved SAP and QA/QC procedures • Acts as a focal point for coordination for quality matters for all project and resolves quality issues • Suspends project activities if quality standards are not maintained • Interfaces with the Navy, including NAVFAC SW QAO, on quality-related items • Discusses and implements Navy technical direction letters related to quality topics with the ARCADIS PM and field team • May appoint a qualified designee(s) to assist him in his responsibilities
Amy Goldberg Day	Project Toxicologist	ARCADIS	• Supports development of the Work Plan and SAP • Provides risk assessment technical oversight
Dennis Dyke	Project Chemist	ARCADIS	• Assists in the development of the SAP • Ensures that sampling personnel have documented training on sampling procedures for specific project requirements • Supports projects as the technical lead for chemical data collection and analysis • Monitors performance of subcontract laboratory and data validator • Audits sample collection activities • Conducts (or assigns a qualified designee to conduct) field QC audits to ensure that project plans are being followed • Validates all field data and coordinates validation of laboratory analytical data • Reviews field and laboratory data against the SAP requirements • Reviews data validation reports • Prepares data quality assessment report to ensure the quality of the data meets the intended use of the data
Emily Sheu	Project Engineer / Field Team Lead	ARCADIS	• Supports development of the Work Plan and SAP • Ensures that all fieldwork is conducted in accordance with the Work Plan and SAP • Provides direction to field staff and subcontractors • Reports field information to ARCADIS PM and Project Chemist

SAP Worksheet #7 – continued

Name	Title/Role	Organizational Affiliation	Responsibilities
Tim Hess	Site Safety and Health Manager	ARCADIS	• Supervises development, implementation, and management of the APP/SSHP • Supervises initial field safety meeting during initiation of the pre-design investigation work • Performs job safety analysis and loss prevention observations during field work implementation
To Be Determined	Wildlife Biologist/ Biological Monitor	To Be Determined	• Implements the biological avoidance, minimization, and mitigation measures specified in the Work Plan • Prepares and conducts the Biological Resource Education Program briefing for contractor and subcontractor personnel

Notes

APP/SSHP	Accident Prevention Plan/Site	Safety and Health Plan
Navy	Department of the Navy	
NAVFAC SW	Naval Facilities Engineering	Command Southwest
PM	Project Manager	
QA/QC	Quality Assurance/Quality Control	
RPM	Regional Project Manager	
SAP	Sampling and Analysis Plan	

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SAP Worksheet #8 — Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel/ Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
No specialized training is required for this project.						

All field personnel who are present at the site are required to meet the health and safety training requirements of Title 29, Code of Federal Regulations, Part 1910.120(e). This includes current Hazardous Waste Operations and Emergency Response Training (40-hour and 8-hour refresher). Copies of field personnel health and safety training records will be maintained in the Project File.

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SAP Worksheet #9 — Project Scoping Session Participants Sheet

Project Name: Remedial Investigation for CTA North and DRMO South **Projected Date(s) of Field Work:** November/December 2013 **PM:** Hoa Voscott	**Site Name:** CTA North, DRMO South **Site Location:** Former Mare Island Naval Shipyard, Vallejo, CA
Date of Session: 03 October 2012 **Scoping Session Purpose:** Project Kickoff Meeting	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Christopher Dirscherl	RPM	BRAC PMO West	(619) 532-0785	christopher.dirscher@navy.mil	RPM
Allan Tamayo	Contract Specialist	BRAC PMO West	(619) 545-6627	allan.tamayo@navy.mil	Contract Specialist
Izzat Amadea	ROICC Representative	BRAC PMO West	(510) 749-5945	izzat.amadea@navy.mil	ROICC Representative
Hoa Voscott	PM	ARCADIS	(510) 596-9609	hoa.voscott@arcadis-us.com	PM
Phil Nicolay	Principal in Charge	ARCADIS	(562) 496-3021	phil.nicolay@arcadis-us.com	Principal in Charge
Judith Nedoff	Principal Scientist	ARCADIS	(510) 596-9523	judith.nedoff@arcadis-us.com	Risk Assessor
Emily Sheu	Staff Engineer	ARCADIS	(510) 596-9653	emily.sheu@arcadis-us.com	Project Engineer

Overview of Meeting

The Project Kickoff Meeting was an internal meeting between the Navy and its contractor, ARCADIS, to make introductions, review the scope of work for the project, assign roles and responsibilities, and discuss the project schedule, communications, quality control, health and safety, project administration, staffing goals, and site access and security. The minutes from this meeting are provided on the following page.

In subsequent informal phone calls and e-mail exchanges, the team discussed the results of the 2012 site inspection and developed the sampling plan described in this SAP.

SAP Worksheet #9 – continued

CTA-N & DRMO-S Remedial Investigation Kickoff Meeting	
MEETING CALLED BY	Hoa Voscott, ARCADIS Project Manager
TYPE OF MEETING	Kickoff Meeting for Contract N62473-11-D-2226, Task Order #0004
MINUTES PREPARED BY	Emily Sheu, ARCADIS
ATTENDEES	Navy: Christopher Dirscherl, Remedial Project Manager Allan Tamayo, Contract Specialist Izzat Amadea, ROICC ARCADIS: Hoa Voscott, Project Manager Phil Nicolay, Principal in Charge Judith Nedoff, Risk Assessor Emily Sheu, Staff Engineer

AGENDA TOPICS

(1) Introductions H. Voscott and C. Dirscherl

DISCUSSION	Hoa and Chris introduced the ARCADIS and Navy teams, respectively. Allan Tamayo was the Contract Specialist representative on the call, but Mark Muralt will be the primary contact for any future questions.
CONCLUSIONS	

ACTION ITEMS	PERSON RESPONSIBLE	DEADLINE

(2) Project Overview H. Voscott

DISCUSSION	Hoa provided a brief summary of ARCADIS' understanding of the project scope (implement RI at CTA-North and DRMO-South sites). Chris provided an update on the PA/SI field work currently underway: currently conducting Geoprobe work in DRMO proper, will move to DRMO-South this week and to CTA-North next week, wrapping up by October 12).
CONCLUSIONS	Sampling scope will be updated and refined based on the PA/SI data.

ACTION ITEMS	PERSON RESPONSIBLE	DEADLINE

SAP Worksheet #9 – continued

(3) Key Team Members H. Voscott

DISCUSSION	Primary CSO contact will be Mel Asuncion.		
CONCLUSIONS			
ACTION ITEMS		PERSON RESPONSIBLE	DEADLINE

(4) Project Schedule H. Voscott

DISCUSSION	The RI WP will cover both sites (separate RI reports will be prepared for each site). Currently there are no plans to sample within 50 feet of the non-tidal wetland area. Chris commented that the DFG will likely have comments on the WP based the Navy's experience with finalizing the PA/SI WP.		
CONCLUSIONS	Internal Draft RI WP will be due 60 days from receipt of the validated PA/SI data. Bonds are due after start of construction (within 15 days from start of construction tasks).		
ACTION ITEMS		PERSON RESPONSIBLE	DEADLINE
Send final PA/SI WP to Hoa		Chris	10/3 - done

(5) Communications H. Voscott

DISCUSSION	Hoa and Chris will establish an informal, weekly update call once the data start coming in.		
CONCLUSIONS			
ACTION ITEMS		PERSON RESPONSIBLE	DEADLINE

(6) Quality Control H. Voscott

DISCUSSION	Izzat had a question regarding the quality control plan and whether it would be a separate document or buried within the SAP/RI WP. Hoa asked for the Navy's preference (currently SAP includes the FSP and QAPP).		
CONCLUSIONS	ARCADIS will ensure that the quality control plan is exhaustive and fulfills all requirements.		
ACTION ITEMS		PERSON RESPONSIBLE	DEADLINE

(7) Health and Safety H. Voscott

DISCUSSION	ARCADIS will prepare the SSHP/APP. ARCADIS will review the safety records of all subcontractors to ensure that they are up to par.
CONCLUSIONS	Izzat can provide ARCADIS with forms for the SSHP and AHAs.

SAP Worksheet #9 – continued

ACTION ITEMS	PERSON RESPONSIBLE	DEADLINE

(8) Project Administration H. Voscott

DISCUSSION	Hoa asked if there was a specific invoice format that should be used. Chris will verify, but thinks that the format should be similar to what ARCADIS has used for Hunters Point/MINS projects in the past.
CONCLUSIONS	

ACTION ITEMS	PERSON RESPONSIBLE	DEADLINE
ARCADIS to provide Chris with examples of the invoice format used for Hunters Point	Hoa/Phil	

(9) Staffing Goals H. Voscott

DISCUSSION	ARCADIS will use Curtis & Tompkins, local business, for laboratory work, and will be reaching out to drillers in the area.
CONCLUSIONS	

ACTION ITEMS	PERSON RESPONSIBLE	DEADLINE

(10) Site Access and Security H. Voscott

DISCUSSION	According to Izzat, there are no access controls at the sites with the exception of the fencing at DRMO-South (the Navy will provide ARCADIS with the lock combination). The bigger issue will be to make sure that equipment left on site is secure. There is a security team that primarily patrols the center and northern parts of Mare Island. ARCADIS may need to coordinate site access with other contractors (Weston working in PWA).
CONCLUSIONS	

ACTION ITEMS	PERSON RESPONSIBLE	DEADLINE
Pre-construction meeting to be held at the site prior to initiating field work	Hoa/Izzat	

SAP Worksheet #10 — Problem Definition

The following sections provide descriptions and background information for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North located at the former Mare Island Naval Shipyard (MINS) in Vallejo California. The Problem Definition is essentially Step 1 of the Data Quality Objectives (DQOs) process (United States Environmental Protection Agency [USEPA] 2006a). Results of the DQO process are discussed in detail in Worksheet #11.

10.1 Site Location and Setting

MINS is located on Mare Island peninsula in southwestern Solano County approximately 25 miles northeast of San Francisco. The peninsula is located within the incorporated boundaries of the City of Vallejo and is bounded by Highway 37 to the north, Carquinez Strait to the south, and the San Pablo Bay to the west (Figure 1). The Napa River (Mare Island Strait) separates Mare Island from the City of Vallejo to the east (Weston Solutions, Inc. [Weston] 2008).

Originally, Mare Island was an island comprised of approximately 1,000 acres of dry land and 300 acres of wetland (Tetra Tech EM, Inc. [TtEMI] 2000). However, land reclamation projects and filling activities have transformed the island into the current peninsula. Mare Island is currently approximately 3.5 miles long by 1.25 miles wide, and occupies approximately 2,800 acres (CH2M Hill 2010).

DRMO South is positioned within the north-central portion of MINS, south of Dump Road and the DRMO site and west of Azuar Drive (Figures 2 and 3; Trevet 2012). CTA North is also located in the north-central portion of MINS. The boundaries of CTA North are roughly defined as the upland area north of Dump Road and west of Azuar Drive (Figure 4). The CTA is not included within the CTA North boundaries (Figure 2); the CTA was transferred to the City of Vallejo, which then transferred the CTA to Mare Island redeveloper, Lennar Mare Island, in 2001.

DRMO South and CTA North were not part of the original Mare Island. These areas were situated below the water line of the San Pablo Bay until between 1911 and 1920 when the area was filled with dredge material removed from Mare Island Strait (Trevet 2012). Additional fill material composed primarily of construction debris and solid waste was deposited within a portion of the CTA North area between 1925 and 1942 (see Figure 4; Trevet 2012).

10.2 Site Description and History

Mare Island was established as a naval shipyard in 1854 following its purchase by the Navy in 1853. The primary mission of MINS was to build, maintain, and repair Navy ships and submarines. The southern portion of the island also served as a munitions storage and production facility (Trevet 2012). MINS operated for 142 years until its closure under the Base Realignment and Closure (BRAC) Program in 1996 (Trevet 2012).

10.2.1 DRMO South

DRMO South is located within the north-central portion of MINS, and part of the site is located within 300 feet of a nontidal wetland area. A large portion of DRMO South is undeveloped while the remainder includes an industrial area and vehicle parking lots. DRMO South is currently vacant, and Section 10.4.1 presents details regarding the historical use of buildings at the site. Removal actions conducted in the neighboring DRMO site between 2005 and 2008 (non-time-critical removal action [NTCRA]) and between 2009 and 2010 (petroleum corrective action [PCA]) included the excavation of small areas of the northwest and eastern boundaries of DRMO South (Figure 3; Trevet 2012).

The NTCRA at DRMO was implemented to address munitions and explosives of concern (MEC) as well as chemicals of potential concern (COPCs), which included metals, semivolatile organic compounds [SVOCs] including polynuclear aromatic hydrocarbons [PAHs], polychlorinated biphenyls [PCBs], and pesticides) located primarily within the Fenced Scrapyard Area (FSA; Weston 2008). The initial phase of the NTCRA involved clearing the upper 18 inches of soil from the FSA within the DRMO. However, during subsequent phases, areas with COPC concentrations exceeding the target cleanup goals, including a small area of the northwest boundary of DRMO South, were over-excavated to a maximum depth of 8.5 feet below ground surface (bgs; Weston 2008).

The PCA was implemented to remove petroleum-impacted soil from: (1) an area within and to the west of Azuar Drive; and (2) an area within and on the north and south sides of Dump Road [Figure 3; Weston 2010]. Portions of the eastern boundary of DRMO South, located adjacent to Azuar Drive, were excavated to depths ranging from 17 to 20 feet bgs; and portions of the site's northern boundary, located adjacent to Dump Road, were excavated to depths ranging from 9 to 19 feet bgs. Confirmation samples indicated that concentrations of total petroleum hydrocarbons (TPH) as Bunker C fuel oil (C12-C40) were less than the Tier 2 screening levels (SLs) for commercial/industrial land use (i.e., 5,000 milligrams per kilogram [mg/kg] for depths between 3 and 10 feet bgs; Weston 2010).

The recent SI sampling, conducted between October and November of 2012, included the collection of 51 soil samples and 11 groundwater samples from 26 borings within the DRMO South area to identify and delineate COPCs in soil and groundwater at the site. Results of the SI are summarized in Section 10.6.1. Historical data are also available from previous investigations conducted at Investigation Area (IA) H2, which included DRMO South, as summarized in the Internal Draft IA H2 RI Report (TtEM 2000).

10.2.2 CTA North

CTA North is located within the north-central portion of MINS. No buildings are or were previously located within the boundaries of CTA North (Figure 4; Trevet 2012). A majority of the site is located near (within 300 feet of) a non-tidal wetland area (Figure 4).

Between 2009 and 2010, petroleum-impacted soil was excavated from an area within and immediately adjacent to CTA North as part of the PCA (Figure 4; Weston 2010). A total of

82,908 bank cubic yards of soil was removed from an approximately 3.7 acre area to depths ranging from 9 to 20 feet bgs (Weston 2010). Confirmation samples were collected and analyzed for TPH as Bunker C fuel oil, and the results were compared with Tier 2 SLs for commercial/ industrial land use. Excavation areas with bottom or confirmation samples exceeding the SLs were over-excavated and resampled with the exception of four sidewall samples located at the northern limit of the excavation area within CTA North (Weston 2010). These samples exhibited TPH concentrations above the Tier 2 SLs but were not over-excavated (Figure 4; Weston 2010).

The recent SI sampling, conducted between October and November of 2012, included the collection of 22 soil samples and 4 groundwater samples from 12 borings within the CTA North area to identify and characterize COPCs in soil and groundwater at the site. Results of the SI are summarized in Section 10.6.1. Historical data are also available from previous investigations conducted at IA H2, which included portions of CTA North, as summarized in the Internal Draft IA H2 RI Report (TtEM 2000).

10.3 Potential Sources

The following sections describe potential or known sources at DRMO South and CTA North based on the results of previous environmental investigations and review of site-related documents and aerial photographs.

10.3.1 DRMO South

Information regarding historical uses of the buildings at DRMO South was presented in the PA/SI Work Plan (Trevet 2012). During site operation, Building 275 was designated as the "Crane Maintenance Shop" and included a paint booth. Hazardous materials (including oils and degreasers) were used and/or stored within this building. Buildings 961 and 965 were used for storage in support of Building 275 operations, and Building 831 was an open-air covered parking structure. A 500-gallon aboveground storage tank located outside of the building perimeter on the northwest side of Building 965 supported an emergency generator. As noted in the Mare Island Health, Safety, and Environmental Building Survey Building 965, dated October 30, 1996, the out-of-service tank was drained of diesel fuel, triple-rinsed, and sent for scrap off site. No containment area related to this tank is present, and there are no visible signs of its presence or past releases. During the 2012 SI, soil boring DRMOS-SB12 was installed in the area of the former tank to characterize the location. No information was available for the other buildings located within DRMO South. Based on aerial photographs, it was determined that the open area west of Building 275 was likely used for equipment storage and vehicle parking (Trevet 2012).

Historical uses of the adjacent DRMO site included: storage of transformers, batteries, and metal scrap; storage of pesticides for maintenance purposes; bailing of paper; handling of petroleum and waste oils; and management of surplus material and scrap from MINS and other military facilities (Weston 2008). A former oil sump box located approximately 50 feet north of Building 661 at DRMO, was determined to be the probable source of petroleum product (Bunker C fuel oil) observed along the west side of Azuar Drive. The oil sump box and associated piping were removed, and portions of Azuar Drive, including those along the eastern boundary of DRMO

South, were excavated as part of the 2009-2010 PCA (see Section 10.2.1; Weston 2010). A portion of the northwestern corner of DRMO South, adjacent to Dump Road, was also excavated during the 2009-2010 PCA (see Section 10.2.1). TPH-contaminated soil in this area was attributed to intermittent and isolated disposal and releases, or backfill with undocumented contaminated materials, e.g., mixed heavy fuel oil and scrap metal (see also Section 10.3.2; CH2M Hill 2009).

Two sites identified in the Internal Draft IA H2 RI Report (TtEM 2000)—Installation Restoration Site 10 (IR10) and Installation Restoration Site 14 (IR14)—were also located in proximity to DRMO South. IR10, also known as Solid Waste Management Unit (SWMU) 107, included the portion of DRMO South located adjacent to (west of) Building 831 as well as an area to the south of the access road that bounds DRMO South to the south. The portion of IR10/SWMU 107 located south of the access road was transferred to the City of Vallejo, and the remaining portion was to be addressed as part of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) work conducted at DRMO South. Historical uses of IR10 included motor vehicle storage and storage of electrical equipment including transformers, rectifiers, and switch gear containing PCBs (TtEM 2000). Storage of electrical equipment is not believed to have been conducted in the portion of IR10/SWMU 107 located within DRMO South.

IR14 comprised the industrial wastewater (IW) collection system that was used to collect, pretreat, and convey wastewater from various sources at Mare Island to the industrial wastewater treatment plant located on the west side of the island (TtEM 2000). The entire IW collection system at Mare Island was composed of approximately 11 IW pump stations and 26,000 linear feet of underground pipeline divided into 11 sections (IW Lines A through K) and installed at approximately 4 to 5 feet bgs. Twenty-five buildings, including an oil reclamation facility, metal cleaning and plating shops, electrical shops, photography laboratories, and paint shops were connected to the IW collection system for wastewater discharge (TtEM 2000). Pump Station 1 (Building 971), which collected wastewater from IW Line B and pumped it into IW Line A, covered an approximately half-acre area to the south of DRMO South and adjacent to Azuar Drive (TtEM 2000). Records indicate that portions of the IW pipeline were repaired over the serviceable life of the IW collection system from 1972 to 1996 (TtEM 2000). Although the exact locations of pipeline leakage and repair were not documented, the most frequent repairs were performed on the IW pipeline at its connections to the pump stations (TtEM 2000).

Potential sources of COPCs at DRMO South include:

- Vehicle (including cranes) storage, equipment storage, maintenance activities, and vehicular traffic,
- Paint booth operations,
- Hazardous materials use/storage,
- Former aboveground storage tank near Building 965,
- Land reclamation/filling activities,

- Former oil sump box and associated piping,
- COPC migration from adjoining areas including the DRMO, IR10/SWMU 107 (the former PCB storage area), and IR14 (Pump Station 1 area), and
- Bedding material in utility corridors.

10.3.2 CTA North

According to the PA/SI Work Plan, a review of site-related documents and aerial photographs did not produce any specific historical uses of CTA North (Trevet 2012). However, portions of the site are located within the 1925-1942 fill material area which is associated with the CTA site (Figure 4). Previous environmental investigations conducted at the CTA revealed elevated PAH, PCB, TPH-d, and/or metals concentrations (i.e., above the screening values) in soil typically at depths shallower than 7 feet bgs (CH2M HILL 2010). The elevated PAH (primarily benzo[a]pyrene [B(a)P]), PCB (Aroclor 1260), and metals (arsenic and lead) concentrations were attributed to past activities conducted within the CTA, including the 1925 to 1942 fill activity, vehicle storage, operation of a classification yard and lumber salvage area, and load testing for shipyard cranes (CH2M HILL 2010).

As discussed in Section 10.2.2, four confirmation sidewall samples collected during the 2009-2010 PCA indicated that TPH was present at concentrations above the Tier 2 SLs within CTA North, at the northern limit of the excavation area along Dump road (Weston 2010). According to the Final Technical Memorandum, Total Petroleum Hydrocarbons at the DRMO Site and Vicinity (CH2M HILL 2009), information obtained during follow-on investigations conducted in 2007 and 2008 demonstrated that the former oil sump box, located approximately 50 feet north of Building 661, was not the source of TPH found along Dump Road. Forensic chemistry findings suggested that the most probable sources of TPH contamination in the Dump Road area included intermittent and isolated disposal and releases, or backfill with undocumented contaminated materials, e.g., mixed heavy fuel oil and scrap metal (CH2M HILL 2009).

Historically, a portion of IR14 (IW) pipeline and sanitary sewer pipeline ran along Dump Road crossing through the southwest corner of CTA North. As part of the 2007 follow-on field investigation, trenches were excavated along this section of co-located IR14 pipeline and sanitary sewer line to determine whether free-phase petroleum hydrocarbons were present and whether the pipelines and their backfill could represent a preferential pathway of migration (CH2M HILL 2009). Free product and/or metal debris were observed in all four trenches at depths from 3 feet bgs to at least 9 feet bgs (CH2M HILL 2009). The southwestern corner of CTA North, including the IR14 and sanitary sewer pipeline area, was excavated as part of the 2009-2010 PCA (see Section 2.2.2). Excavation was completed to at least 10 feet bgs in this area.

In addition, a portion of storm sewer pipeline ran along Azuar Drive adjacent to the CTA and part of CTA North as well as the DRMO. According to the Final PCA Completion Report (Weston 2010), the storm sewer line parallel to Azuar Drive in this area was removed as part of the 2009-2010 PCA, and a replacement line was installed along the west side of Azuar Drive.

Potential sources at CTA North include:

- Historical activities conducted within the adjacent CTA,
- Crane storage and operation, operation of a classification yard and lumber salvage area, and vehicle storage,
- Contaminated (1925 to 1942) fill material,
- COPC migration from adjoining areas including the CTA, DRMO, and DRMO South,
- Bedding material in utility corridors, and
- IR14 (IW) and sanitary sewer pipelines.

10.4 Ecological Setting

Ecological habitat at DRMO South and CTA North consists of upland habitat. Uplands are well-drained areas above the effects of tidal action (TtEM 2000). At Mare Island, uplands typically consist of grasslands and shrublands with little native vegetation remaining located along the boundary of non-tidal wetland areas (TtEM 2000). The vegetation at DRMO South and CTA North primarily includes ruderal grasses and other opportunistic plants (TtEM 2000).

Adjacent non-tidal wetland habitat contains a variety of vegetation, including cattails, pickleweed, and sedges (TtEM 2000). These areas are commonly characterized by standing water during the winter and spring and are dry during the summer and fall (TtEMI 2000). The non-tidal wetland habitat is likely to support various species of invertebrates, birds, and small mammals. The upland habitat may also support some of these species.

10.5 Existing and Potential Future Land Use

Historically, DRMO South has been used for industrial activities though a large portion of the site is open space. Per the current reuse plan, future uses of DRMO South include light industrial and open space (City of Vallejo 2007). CTA North has been undeveloped open space and will remain as such per the current reuse plan (City of Vallejo 2007).

10.6 Conceptual Site Model

This preliminary conceptual site model (CSM) describes current understanding of the relationship between chemical sources, migration pathways, and exposure routes to potential receptors based on the 2012 SI results and review of site-related historical documents. This CSM will be updated and refined based on the results of the RI field work to be conducted under this SAP.

10.6.1 Nature and Extent of Affected Media

The following sections describe the nature and extent of affected soil and groundwater at DRMO South and CTA North based on the results of the 2012 SIs and previous investigations conducted at IA H2.

10.6.1.1 DRMO South

Soil:

PCBs, petroleum hydrocarbons, PAHs, metals, pesticides, and volatile organic compounds (VOCs) have been detected in soil at DRMO South. Constituents detected at concentrations above the screening criteria—referenced below and summarized in the Preliminary Assessment/Site Inspection (PA/SI) Work Plan (Trevet 2012)—during the 2012 SI included PCBs, TPH-diesel (TPH-d) and TPH-motor oil (TPH-mo), PAHs, and metals.

PCBs:

Results of the 2012 SI indicated that Aroclor 1260 was detected in 16 out of 51 samples at concentrations ranging from 0.021 mg/kg (estimated value) to 0.97 mg/kg (DRMOS-SB21 at 1 foot bgs). The maximum detected concentration of Aroclor 1260 exceeded the 2013 United States Environmental Protection Agency (USEPA) industrial Regional Screening Level (RSL) of 0.74 mg/kg. However, the 95 percent upper confidence limit (UCL) of the mean for Aroclor 1260, calculated using USEPA ProUCL software, was 0.153 mg/kg, below both the industrial and residential RSLs. The maximum detected concentration of Aroclor 1260 was below the ecological screening level for PCBs of 40 mg/kg (Oak Ridge National Laboratory [ORNL] screening levels specific to terrestrial plants). No other Aroclors were detected in soil.

Review of the IA H2 RI data indicated that the results of historical PCB sampling conducted within the DRMO South area were generally consistent with the 2012 SI results. The exception was the relatively elevated concentration of Aroclor 1260 detected at one 1994 sample location near the site's southern boundary (Figure 3), within the portion of DRMO South that overlaps with IR10 (the Building 831, former PCB storage area). A soil sample collected from 2.5 feet bgs at this location had an estimated Aroclor 1260 concentration of 2 mg/kg, above the Mare Island PCB cleanup criterion of 1 mg/kg for residential land use (TtEM 2000).

Petroleum Hydrocarbons:

Results of the 2012 SI indicated that TPH-d was detected at concentrations ranging from 4.2 mg/kg (estimated value) to 450 mg/kg, while TPH-mo was detected at concentrations ranging from 14 mg/kg (estimated value) to 1,600 mg/kg. TPH-d and/or TPH-mo concentrations at seven boring locations exceeded the 2013 shallow soil ESLs for residential use where groundwater is not a source of drinking water (100 mg/kg for middle distillates [diesel] and 500 mg/kg for residual fuels [motor oil]). As shown on Figure 3, these borings were located within the industrial area of the site. Reported TPH-d and TPH-mo concentrations did not exceed the 2013 Regional Water Quality Control Board (Water Board) shallow soil Environmental Screening Levels (ESLs) for industrial use (500 mg/kg for middle distillates and 2,500 mg/kg for residual fuels).

Review of the IA H2 RI data indicated that the results of historical TPH sampling conducted within the DRMO South area were generally consistent with the 2012 SI results. Samples with elevated TPH concentrations above the screening criteria were primarily located within the

industrial areas of the site. The exception was the elevated concentrations of TPH-d (estimated 1,290 mg/kg at 0 feet bgs and estimated 1,030 mg/kg at 3 feet bgs) detected at one boring advanced in the northwestern upland area of DRMO South (Figure 3). A cluster of samples with elevated TPH-d concentrations above the residential and/or industrial ESLs at depths of up to 14.5 feet bgs was also observed in the southeastern corner of DRMO South near the Pump Station 1 area (Figure 3).

Diesel generally consists of hydrocarbons in the carbon range of C10 to C28, while motor oil varies and can include hydrocarbons with carbon numbers ranging from 18 (C18) to greater than 36 (C36+). In comparison, the product targeted during the 2009-2010 PCA (see Section 10.2.1) demonstrated a hydrocarbon pattern similar to that of Bunker C fuel oil (i.e., C12 to C40), i.e., samples contained both diesel-range hydrocarbons and 'heavier-end' hydrocarbons in the motor oil range.

PAHs:

Results of the 2012 SI indicated that the B(a)P-equivalent concentration for total carcinogenic PAHs at the site (maximum of 0.668 mg/kg in the sample collected from 7 feet bgs at DRMOS-SB20) was below the background level of 0.92 mg/kg (PG&E and Navy 2002). This suggests that PAH concentrations at DRMO South are associated with background levels in northern California surface soil. PAHs were also evaluated individually in comparison with the 2013 USEPA industrial RSLs. B(a)P was detected at concentrations above or equal to the 2013 USEPA industrial RSL of 0.21 mg/kg at eight boring locations (see Figure 3). A majority of these borings were located within the industrial area of the site; however, one boring (DRMOS-SB02) was located in the undeveloped area south of the NTCRA area boundary (Figure 3). The remaining PAHs were not detected at concentrations exceeding their respective USEPA industrial RSLs.

Metals:

Results of the 2012 SI indicated that arsenic was the only metal detected in soil at concentrations above the corresponding screening level benchmark for the industrial exposure scenario: the 2013 USEPA industrial RSL of 2.4 mg/kg. Arsenic was detected at concentrations ranging from 3.04 mg/kg to 57.3 mg/kg (DRMOS-SB02 at 8 feet bgs) with a 95 percent UCL of 19.3 mg/kg. The background concentration of arsenic in fill material is 36 mg/kg (TtEMI 2002). Only three out of 51 detections exceeded this background value: 57.3 mg/kg (DRMOS-SB02 at 8 feet bgs), 39.3 mg/kg (DRMOS-SB03 at 8 feet bgs), and 37.2 mg/kg (DRMOS-SB11 at 7 feet bgs). The distribution of these locations (e.g., not clustered; see Figure 3) did not indicate a pattern or specific area of elevated arsenic. In addition, elevated (above background) arsenic levels were not restricted to near surface soil, indicating that the source is likely ambient concentrations of arsenic in dredge spoils used to develop the area.

Review of the IA H2 RI data indicated that lead has also been detected at DRMO South at concentrations above both its background level in fill material (59 mg/kg; TtEM 2002) and its industrial screening level benchmark (industrial California Human Health Screening Level

[CHHSL] of 320 mg/kg). As shown in Figure 3, elevated lead was primarily detected in samples collected from the southeastern corner of DRMO South, near the Pump Station 1 area, at up to 13.5 feet bgs.

Based on the 2012 SI results, maximum detected concentrations of arsenic, barium, cobalt, copper, lead, manganese, nickel, selenium, and zinc exceeded both background levels and the ecological screening benchmarks (USEPA Interim Ecological Soil Screening Levels [EcoSSLs] specific to soil invertebrates or terrestrial plants). No ecological screening criteria were available for chromium; however, the maximum detected concentration of chromium (286 mg/kg) was above background 140 mg/kg; TtEMI 2002). This suggests that chromium concentrations in soil at DRMO South may not be attributed to background and further evaluation may be warranted.

Groundwater:

Petroleum hydrocarbons, PAHs, metals, pesticides, and VOCs have been detected in groundwater at DRMO South. Constituents detected at concentrations above the screening criteria—referenced below and summarized in the PA/SI Work Plan (Trevet 2012)—during the 2012 SI included PAHs, metals, pesticides, and VOCs.

PAHs:

Results of the 2012 SI indicated that naphthalene was reported in eight groundwater samples at concentrations ranging from 0.31 micrograms per liter (µg/L, estimated value) to 0.98 µg/L (estimated value at DRMOS-SB03), above the residential tap water RSL of 0.14 µg/L. However, use of site groundwater for municipal and/or domestic supply is considered unlikely at DRMO South.

Metals:

Results of the 2012 SI indicated that arsenic, molybdenum, and nickel were detected in groundwater at concentrations above background.

- Arsenic detections ranged from 0.976 µg/L to 325 µg/L (DRMOS-SB24). The background arsenic concentration in groundwater is 78 µg/L (TtEMI 2002) and the drinking water RSL is 0.045 µg/L. Three of the 11 detections were greater than the background value of 78 µg/L: 325 µg/L (DRMOS-SB24), 128 µg/L (DRMOS-SB15), and 175 µg/L (DRMOS-SB04). These locations did not correspond with the locations where arsenic was observed above background in subsurface soil (see Soil discussion above).
- Maximum detected concentrations of molybdenum and nickel were below the residential human health screening criteria (tap water RSLs).

Based on 2012 SI results, maximum detected concentrations of arsenic and nickel were above the ecological screening benchmarks (USEPA National Recommended Ambient Water Quality Criteria [AWQC], criteria continuous concentrations for marine environments). Ecological screening criteria for inorganics in water were considered due to the proximity of DRMO South

to non-tidal wetland habitat. For the purpose of intial screening, it was conservatively assumed that groundwater could surface in the nearby wetlands and be available to aquatic marine species.

Pesticides:

Results of the 2012 SI indicated that endrin was the only organochlorine pesticide detected in the groundwater. Endrin was detected in one sample (the field duplicate collected from DRMOS-SB13) at an estimated concentration of 0.026 µg/L, lower than the residential tap water RSL of 1.7 µg/L but greater than the ecological screening benchmark of 0.0023 µg/L (AWQC, criterion continuous concentration for marine environments).

VOCs:

Results of the 2012 SI indicated that chloroform was detected in one sample (DRMOS-SB11) at an estimated concentration of 1.0 µg/L, above the residential tap water RSL of 0.19 µg/L. Chloroform is a common laboratory contaminant when analyzing for VOCs.

10.6.1.2 CTA North

Soil:

PCBs, petroleum hydrocarbons, PAHs, metals, pesticides, VOCs, and SVOCs have been detected in soil at CTA North. Constituents detected at concentrations above the screening criteria—referenced below and summarized in the PA/SI Work Plan (Trevet 2012)—during the 2012 SI included PCBs, TPH-d and TPH-mo, PAHs, and metals.

PCBs:

Results of the 2012 SI indicated that Aroclor 1260 was detected in 8 out of 22 samples at concentrations ranging from 0.027 mg/kg (estimated value) to 0.39 m/kg (CTAN-SB09 at 1 foot bgs). The maximum detected concentration of Aroclor 1260 exceeded the 2013 USEPA residential RSL of 0.22 mg/kg but was below the industrial RSL of 0.74 mg/kg. The 95 percent UCL was 0.129 mg/kg, below the residential RSL of 0.22 mg/kg. The maximum detected concentration of Aroclor 1260 was well below the ecological screening level of 40 mg/kg (ORNL screening levels specific to terrestrial plants). No other Aroclors were detected in soil.

Petroleum Hydrocarbons:

Results of the 2012 SI indicated that TPH-d was detected at concentrations ranging from 5.0 mg/kg (estimated value) to 140 mg/kg, while TPH-mo was detected at concentrations ranging from 17 mg/kg (estimated value) to 5,400 mg/kg. TPH-d and/or TPH-mo were detected at three boring locations (CTAN-SB03, CTAN-SB06, and CTAN-SB09; see Figure 4) at concentrations exceeding the 2013 shallow soil ESLs for residential use where groundwater is not a source of drinking water (100 mg/kg for middle distillates [diesel] and 500 mg/kg for residual fuels [motor oil]). The concentration of TPH-mo detected at CTAN-SB03 (5,400 mg/kg at 1 foot bgs) also

exceeded the 2013 shallow soil ESL for industrial use of 2,500 mg/kg (residual fuels). CTAN-SB06 and CTAN-SB09 are both located within the 1925-1942 fill area. CTAN-SB03 was located near a utility corridor, and elevated TPH-d and TPH-mo concentrations above the screening criteria were only detected in the shallow soil sample (1 foot bgs) collected at this location.

Review of the IA H2 RI data indicated that the results of historical TPH sampling conducted within CTA North were generally consistent with the 2012 SI results. Samples with elevated TPH-d concentrations above the screening criteria were located within the 1925-1942 fill area (shallow soil) and near the PCA area boundary (at a depth of up to 8.5 feet bgs).

Diesel generally consists of hydrocarbons in the carbon range of C10 to C28, while motor oil varies and can include hydrocarbons with carbon numbers ranging from 18 (C18) to greater than 36 (C36+). In comparison, the product targeted during the 2009-2010 PCA (see Section 10.2.2) demonstrated a hydrocarbon pattern similar to that of Bunker C fuel oil (i.e., C12 to C40), i.e., samples contained both diesel-range hydrocarbons and 'heavier-end' hydrocarbons in the motor oil range.

PAHs:

Results of the 2012 SI indicated that the B(a)P-equivalent concentration for total carcinogenic PAHs at the site exceeded the background level of 0.92 mg/kg (PG&E and Navy 2002). This suggests that PAH concentrations at CTA North may not be attributable to ambient levels in northern California surface soil. PAHs were also evaluated individually in comparison with the 2013 USEPA industrial RSLs. B(a)P was detected at concentrations above or equal to the 2013 USEPA industrial RSL of 0.21 mg/kg at four boring locations (see Figure 4). Dibenzo(a,h)anthracene was also detected at one of these locations (CTAN-SB09 at 1 foot bgs) at a concentration of 0.24 mg/kg, above the industrial RSL of 0.21 mg/kg. The maximum detected concentrations of B(a)P (0.84 mg/kg and 0.68 mg/kg) occurred at CTAN-SB09 where many of the maximum detected metals concentrations were also observed, indicating a potential source area.

Metals:

Results of the 2012 SI indicated that arsenic, lead, and mercury were the only metals that were detected at concentrations greater than the screening-level benchmark for the industrial exposure scenario:

- Arsenic detections ranged from 6.75 mg/kg to 251 mg/kg (CTAN-SB09 at 1 foot bgs). The background concentration of arsenic in fill material is 36 mg/kg (TtEMI 2002). As shown in Figure 4, arsenic concentrations in four samples collected from two boring locations (CTAN-SB06 and CTAN-SB09, both located within the 1925-1942 fill material area) exceeded the background value.

- Lead detections ranged from 23.5 mg/kg to 23,200 mg/kg (CTAN-SB06 at 2.5 feet bgs). The background value for lead in fill material is 59 mg/kg (TtEMI 2002) and the industrial CHHSL for lead is 320 mg/kg. As shown in Figure 4, lead concentrations in six soil samples collected from four boring locations (CTAN-SB05, CTAN-SB06, CTAN-SB08, and CTAN-SB09) located within the 1925-1942 fill material area exceeded the industrial CHHSL. Lead concentrations in five of the six soil samples also exceeded the sum of the industrial CHHSL plus background value, 379 mg/kg. These included the samples collected from 1 and 2.5 feet bgs at CTAN-SB06, the sample collected from 1 foot bgs at CTAN-SB08, and the samples collected from 1 and 2 feet bgs at CTAN-SB09.
- Mercury detections ranged from 0.0716 mg/kg (estimated value) to 80 mg/kg (CTAN-SB09 at 1 foot bgs). The background value for mercury in fill material is 2 mg/kg (TtEMI 2002) and the industrial RSL for mercury is 43 mg/kg. The mercury concentration at one location (CTAN-SB09) located within the 1925-1942 fill material area exceeded the industrial RSL.

Based on the 2012 SI results maximum detected concentrations of arsenic, copper, lead, manganese, mercury, molybdenum, nickel, vanadium, and zinc were above both their background levels and the ecological screening benchmarks (EcoSSLs or ORNLs). Maximum detected concentrations of barium, cobalt, and selenium exceeded the ecological screening benchmarks (EcoSSLs) but background levels were not available.

Groundwater:

Petroleum hydrocarbons, PAHs, metals, and VOCs have been detected in groundwater at CTA North. Constituents detected at concentrations above the screening criteria—referenced below and summarized in the PA/SI Work Plan (Trevet 2012)—during the 2012 SI included PAHs and metals.

PAHs:

Results of the 2012 SI indicated that the maximum and only reported concentration of naphthalene was 0.62 µg/L (estimated value) at CTAN-SB10. This detection exceeded the residential tap water RSL of 0.14 µg/L. However, use of site groundwater for municipal and/or domestic supply is considered unlikely at CTA North.

Metals:

Results of the 2012 SI indicated that only molybdenum and nickel were detected in groundwater at concentrations greater than background:

- Nickel was detected at concentrations ranging from 10.6 µg/L (CTAN-SB12) to 59.2 µg/L (CTAN-SB08), above the ecological screening benchmark of 8.2 µg/L (AWQC, criterion continuous concentration for marine environments) but below the residential tap water RSL of 300 µg/L. Ecological screening criteria for inorganics in water were

considered due to the proximity of CTA North to non-tidal wetland habitat. For the purpose of intial screening, it was conservatively assumed that groundwater could surface in the nearby wetlands and be available to aquatic marine species.

- Molybdenum was detected at estimated concentrations ranging from 3.31 µg/L (CTAN-SB12) to 9.15 µg/L (CTAN-SB08), below both human health and ecological screening criteria.

10.6.2 Potential Release Mechanisms and Transport Pathways

Potential sources of COPCs identified at DRMO South and CTA North are discussed in Section 10.3.1 and 10.3.2, respectively.

Potential release mechanisms associated with these sources include:

- Past spills, leaks, or releases from hazardous materials, equipment, and/or vehicle storage and handling areas,
- Historical handling and disposal practices at Mare Island, and
- COPC migration from offsite areas;

Primary potential migration pathways include:

- Wind erosion and transport,
- Surface-water runoff erosion and transport to the non-tidal wetland area,
- Volatilization from soil or groundwater to soil vapor and the atmosphere,
- Migration to groundwater,
- Transport in groundwater, and
- Transport through preferential pathways (e.g., active or abandoned utility pipelines, more permeable utility line backfill, and the debris layer).

10.6.3 Potential Receptors and Exposure Pathways

Per the current reuse plan, future uses of DRMO South include light industrial and open space while CTA North is anticipated to remain as undeveloped open space (City of Vallejo 2007). Given these future use scenarios, potential receptors are limited to recreational users, construction workers (assuming some redevelopment and/or restoration of the land will occur), commercial/industrial workers (portions of DRMO South only), and ecological receptors. The most sensitive human health exposure scenario (i.e., residential exposure) is also evaluated to establish the baseline for unrestricted release of the property and assess whether land use restrictions may be necessary.

An exposure pathway is a mechanism by which receptors may come into contact with site-related chemicals. The USEPA (1989) describes a complete exposure pathway in terms of four components:

- A source and mechanism of chemical release
- A retention or transport medium
- A receptor at a point of potential exposure to a COPC
- An exposure route at the exposure point

If any of these four components is not present, then a potential exposure pathway is considered incomplete and is not evaluated further in a risk assessment. If all four components are present, a pathway is considered complete. Some pathways, although complete, can be considered insignificant if they are likely to contribute only a small fraction of the total exposure/dose.

Potentially complete exposure pathways for human receptors are discussed below and summarized on Figure 5:

- After development at the site, the surface soil may be mildly disturbed or possibly graded with subsurface soil. Commercial/industrial workers, recreational users, and resident adults and children may be exposed to surface soil (0 to 0.5 feet bgs) and subsurface soil (0 to 10 feet bgs) via incidental soil ingestion, soil particulate inhalation, and direct dermal contact.
- During development of the site, construction workers may be exposed to surface and subsurface soil via incidental soil ingestion, soil particulate inhalation, and direct dermal contact. Maintenance/utility workers performing trenching or other maintenance activities may also be exposed to surface and subsurface soil via incidental soil ingestion, soil particulate inhalation, and direct dermal contact.
- Groundwater at the site is not anticipated to be used for domestic or industrial purposes. However, to support the risk management decision process, COPCs in groundwater will be compared to applicable screening criteria for residential use.
- Due to the relatively shallow groundwater at the site, trenching activities may lead to some pooling of groundwater, which may result in exposure via direct dermal contact to construction or maintenance/utility workers. However, dewatering activities are anticipated to be implemented if groundwater does infiltrate into the trenches, minimizing the likelihood of direct contact to the trench worker.
- Volatile compounds have only been detected infrequently in soil and groundwater and at relatively low concentrations. While the inhalation of volatile compounds in ambient air would likely not contribute significantly to cumulative risk at DRMO South and CTA North, it will be considered a potentially complete exposure pathway for all human receptors. Exposure to volatiles in indoor air will be evaluated if volatiles are identified in the subsurface adjacent to buildings.

Potentially complete exposure pathways for ecological receptors include wildlife and plant exposures to surface soil (0 to 2 feet bgs) via direct contact and/or ingestion, plant exposures to shallow soil (0 to depth of first groundwater or 6 feet bgs, whichever is shallower) and shallow groundwater, and ingestion of food items (i.e., plant and/or animal tissue) by wildlife. Although

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burrowing organisms may contact deeper soils (up to 6 feet bgs), burrowing in saturated soils does not occur. Therefore, shallow soil from 0 to 6 feet bgs will be evaluated for burrowing receptors only if the depth to groundwater is greater than 6 feet bgs.

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SAP Worksheet #11 — Project Quality Objectives/Systematic Planning Process Statements

This worksheet documents the development of project quality objectives (PQOs) for the remedial investigation (RI) at DRMO South and CTA North using the United States Environmental Protection Agency (USEPA) Data Quality Objectives (DQO) process. The PQOs are project-specific DQOs.

The seven steps of the USEPA DQO process are as follows (USEPA 2006a):

1. State the problem
2. Identify the goals of the study
3. Identify information inputs
4. Define the boundaries of the study
5. Develop the analytic approach
6. Specify performance or acceptance criteria
7. Develop the plan for obtaining data

General discussions of each step of the DQO process are presented below.

11.1 State the Problem

The objectives of this investigation are as follows:

- To further evaluate and/or delineate potential chemicals of potential concern (COPCs) identified in soil and groundwater at DRMO South and CTA North during the site inspections (SIs) conducted in October and November of 2012 and during previous investigations. Note that one objective of the 2012 SIs, as stated in the Preliminary Assessment/Site Inspection Work Plan, was to evaluate the extent of elevated concentrations of petroleum hydrocarbons (i.e., as Bunker C fuel oil) in soil near the 2009-2010 Petroleum Corrective Action (PCA) excavation area on the southern boundary of CTA North (Trevet 2012). Samples collected from three boreholes (CTAN-SB11, CTAN-SB12, and CTAN-SB13) advanced near the northern limit of the excavation satisfied this objective. These samples did not exhibit elevated petroleum hydrocarbon concentrations above the screening criteria (see Section 10.6.1.2).
- To determine whether COPCs identified in soil and groundwater at the sites would result in unacceptable risks to future receptors.

Refer to Worksheet #10 for additional details. Potential COPCs included those chemicals that were detected at concentrations above the applicable ecological and/or human health screening criteria in soil and/or groundwater at DRMO South or CTA North during the SI. These included:

- Polychlorinated biphenyls (PCBs; specifically Aroclor 1260), total petroleum hydrocarbons as diesel (TPH-d), total petroleum hydrocarbons as motor oil (TPH-mo), polycyclic aromatic hydrocarbons (PAHs), and metals in soil at DRMO South;

- PAHs, metals, pesticides (endrin), and volatile organic compounds (chloroform) in groundwater at DRMO South;
- PCBs (Aroclor 1260), TPH-d, TPH-mo, PAHs, and metals in soil at CTA North; and
- PAHs and metals in groundwater at CTA North.

Note that the TPH-d and TPH-mo analyses are anticipated to cover the hydrocarbon range observed for the product that was targeted during the 2009-2010 PCA (i.e., C12 to C40, Bunker C fuel oil).

In general, potential COPCs were selected for further sampling as part of the RI field work if:

- They were considered to be significant risk drivers given anticipated future land use at the sites (light industrial and open space at DRMO South, and open space at CTA Norht);
- Distribution of the exceedance locations (i.e., sample locations having concentrations exceeding the screening criteria) indicated a potential source that could be further delineated (e.g., 1925-1942 fill material area; COPCs associated with the adjoining remedial action areas at the DRMO and CTA sites; releases from storage tanks, vehicles, or equipment); and
- Additional data were required to assess potential migration pathways or complete a defensible risk risk assessment (e.g., a minimum of six samples is usually recommended for calculating exposure point concentrations for risk assessment purposes).

11.2 Identify the Goals of the Study

The primary decision questions for the RI are:

1. In the DRMO South and CTA North, are concentrations of chemicals in soil at the proposed sampling locations (Figures 3 and 4) below the project action limits (PALs) as listed in Worksheet #15?
2. In CTA North, are concentrations of chemicals in groundwater at the proposed sampling locations (Figure 3) below PALs as listed in SAP Worksheet #15?
3. Do baseline human health and ecological risk assessment results indicate that these chemicals pose an unacceptable risk to human health?

Results from the RI and Navy recommendations for the sites will be presented to the regulatory agencies for their review and concurrence. In addition to direct comparison of analytical results to PALs, a weight-of-evidence approach will be used to derive the recommendations. Section 11.5 presents the decision rules to be used when evaluating the data.

11.3 Identify Information Inputs

Inputs necessary to resolve the decision questions include the following information resources.

- Proposed sampling locations for this RI
- Screening criteria for soil and groundwater as listed in Worksheet #15
- Validated soil and groundwater data collected previously (e.g., during the SI) and during this RI
- Analytical methods and analytes for each sampling matrix as listed on Worksheet #15

11.4 Define the Boundaries of the Study

11.4.1 Lateral Boundaries

In general, the study will cover the DRMO South site (approximately 15 acres) and the CTA North site (approximately 6 acres). No samples will be collected from within the footprints of the 2009-2010 petroleum corrective action area or the 2005-2008 non-time critical removal action area as it is assumed that soil in these areas is clean fill material. In addition, no samples will be collected from within 50 feet of the nontidal wetlands. The proposed soil and groundwater sampling locations define the exact lateral boundaries for this study, and these are shown on Figures 3 and 4. Detailed rationale for the sampling locations is discussed in SAP Worksheet #17. In general, the locations (and depths) of the proposed samples were chosen based on the preliminary conceptual site model to: (1) confirm and/or further delineate areas of potential COPC impacts identified through review of the 2012 SI data and historical Investigation Area H2 RI data; and (2) fill in data gap areas.

11.4.2 Vertical Boundaries

Soil samples will be collected from two or more depth intervals at each boring location. Soil samples will be collected from depths of up to 15 feet below ground surface (bgs) at DRMO South and up to 10 feet bgs at CTA North based on the vertical extent of impacts observed during previous investigations. The depths of the deeper samples (i.e., samples collected from below 0.5 feet bgs) may be biased if visual observations and/or photoionization detector screening of the soil column indicate the possible presence of COPCs.

Groundwater samples will be collected based on the observed depth of the water table and the final screening interval. In general, groundwater samples will be collected from the uppermost water-bearing zone.

11.4.3 Temporal Boundaries

The proposed fieldwork schedule is detailed on SAP Worksheet #16.

11.5 Develop the Analytical Approach

1. If chemicals in soil samples are below PALs (Worksheet #15) and risk assessment results indicate no unacceptable risk to potential receptors, then no further action will be

recommended for soil at the site. Otherwise, additional action will be discussed with the BRAC Cleanup Team (BCT).

2. If chemicals in groundwater samples are below PALs (Worksheet #15) and risk assessment results indicate no unacceptable risk to potential receptors, then no further action will be recommended for groundwater at the site. Otherwise, additional action will be discussed with the BCT.

11.6 Specify Performance or Acceptance Criteria

Two sources of decision errors have been identified: sampling design and measurement errors. Sampling design errors are a function of the selection of sample locations or analytical methods used to characterize the site to be studied. Measurement errors are a function of the procedures used to collect and analyze the samples. Decision errors will be limited by a careful evaluation of the data and adherence to established data collection procedures.

The sampling design for the pre-design investigation is judgmental rather than probability-based. Consequently, statistical inference techniques to precisely quantify uncertainty are not applicable, and no statistical limits on uncertainty were specified. In accordance with USEPA guidance (USEPA 2002), a judgmental sampling design is appropriate for investigations where reliable historical and physical knowledge of the feature or condition already exists. The tolerable limits on decision errors associated with statistical sampling designs do not apply directly to judgmentally located samples. Sampling design error was controlled by careful planning conducted in consultation with Federal Facility Site Remediation Agreement stakeholders and using the professional judgment of project personnel to achieve the study goals.

Measurement errors that arise during the various steps of the sample-measurement process (e.g., sample collection, sample handling, sample preservation, sample analysis, data reduction, and data handling) are possible regardless of the sample design. Neither measurement errors nor variability can be eliminated, but they can be measured and evaluated with appropriate procedures. The analytical methods, method reporting limits, and project-specific groundwater screening criteria for soil and groundwater samples are discussed in Worksheet #15. Measurement error is further managed by using standard procedures, reviewing data records, and managing data quality.

11.7 Develop the Plan for Obtaining Data

The sampling plan was developed based on the study goals, available chemical data, and analytical approach described in Sections 11.2, 11.3, and 11.5 above. RI sampling will consist of the following:

- Collection of 48 soil samples from 17 sampling locations at DRMO South. Soil samples will be analyzed for metals, PAHs, PCBs, and/or TPH-extractable (TPH-e, which includes TPH-d and TPH-mo). A detailed list of analytes is provided in Worksheet #15.

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- Collection of four groundwater samples from four temporary monitoring well locations at DRMO South. Groudnwater samples will be analyzed for metals, PAHs, PCBs, and/or TPH-e.
- Collection of 39 soil samples from 12 sampling locations at CTA North. Soil samples will be analyzed for PAHs, PCBs, TPH-e (which includes TPH-d and TPH-mo), and/or metals. A detailed list of analytes is provided in Worksheet #15.
- Collection of three groundwater samples from three sampling locations at CTA North. Groundwater samples will be analyzed for PAHs, PCBs, TPH-e, and/or metals. A detailed list of analytes is provided in Worksheet #15.

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SAP Worksheet #12 — Measurement Performance Criteria Table

Measurement Performance Criteria Table – Field QC Samples (Soil)

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)
Equipment Rinsate	All	1 per day for all reusable sampling equipment	Accuracy/Bias – Contamination	No analyte > LOQ	S&A
Source Blank	All	1 per lot of water used to collect equipment rinsate samples	Accuracy/Bias – Contamination	No analyte > LOQ	S&A
Temperature Blank	See Note 1	1 per sample cooler	Representativeness	2 to 6ºC	S&A

Notes

1 Temperature blanks are used to determine whether samples have been adequately cooled during shipment and storage. A temperature blank will be provided with each sample cooler and tested upon arrival at the laboratory to ensure that the temperature of the contents of the cooler meets the performance criteria.

2 Field duplicates will not be collected for soil samples during this project due to the heterogeneous nature of the soil matrix.

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Measurement Performance Criteria Table – Field QC Samples (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)
Equipment Rinsate	All	1 per day for all reusable sampling equipment	Accuracy/Bias – Contamination	No analyte > LOQ	S&A
Source Blank	All	1 per lot of water used to collect equipment rinsate samples	Accuracy/Bias – Contamination	No analyte > LOQ	S&A
Field duplicate	All	10% or 1 per 10 groundwater samples	Precision	RPD <25%	S
Temperature Blank	All	1 per sample cooler	Representativeness	2 to 6ºC	S&A

Notes:

>	greater than
ºC	degrees Celsius
DoD	Department of Defense
DQI	data quality indicator
LOQ	limit of quantitation
QC	quality control
S	sampling
A	analytical

SAP Worksheet #13 — Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generators (originating organization, data types, data generation/collection dates)	How Data Will Be Used	Limitations on Data Use
Validated soil and groundwater data from previous investigations	Trevet, Preliminary Assessment/Site Inspection for Defense Reutilization and Marketing Office South and Crane Test Area North	Trevet; Soil and groundwater data; October and November 2012	Aid to identify analytes of concern and sample locations	None
Soil and groundwater data from previous investigations	Tetra Tech EM, Inc., *Internal Draft Investigation Area H2 Remedial Investigation, Mare Island, Vallejo, California*, December 2000	Tetra Tech EM, Inc.; Soil and groundwater data; Prior to 2000	Aid to identify analytes of concern and sample locations	None
Site information and description of previous investigative procedures	Trevet, *Final Preliminary Assessment/Site Inspection Work Plan, Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California*, September 2012	Trevet; Site information and description of proposed investigative procedures; September 2012	For background information on the sites, current understanding of the conceptual site models for the sites, and descriptions of the proposed investigative procedures	None

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SAP Worksheet #14 — Summary of Project Tasks

This worksheet summarizes the project tasks for this investigation. Project tasks include:

- Utility clearance
- Site preparation
- Soil sample collection
- Monitoring well installation and sampling
- Temporary well abandonment
- Land survey
- Decontamination
- Investigation-derived waste (IDW) management
- Data management and review (including third-party validation)

Specific procedures to be followed during field activities are detailed in the sections below.

14.1 Utility Clearance

ARCADIS will review available records (e.g., site plans, utility maps, as-built drawings) to screen proposed sample locations for subsurface features prior to drilling. Underground Service Alert North will be contacted at least 72 hours prior to initiating intrusive activities to locate known underground utilities in the areas where drilling is planned. In addition, a geophysical subcontractor will be retained to locate subsurface utilities using magnetic and/or electromagnetic methods. The geophysical subcontractor will mark the locations of detectable subsurface utilities and/or obstructions in and around the proposed sample locations, within a 5-foot radius.

14.2 Site Preparation

Planned preparatory activities include notification of appropriate personnel and a field visit to identify and mark the locations of the proposed samples and confirm that they are accessible. The proposed sample locations will be identified using a hand-held Global Positioning System unit and marked using spray paint or stakes. A biological monitor will accompany the project team on the field visit to provide guidance on acceptable routes of ingress and egress. The biological monitor will also ensure that proposed sample locations are not within 50 feet of the non-tidal wetland. If the biological monitor determines that a proposed location is within 50 feet of the non-tidal wetland, the location will be shifted such that it is no longer within the buffer zone. The RI field work will be coordinated with the Resident Officer in Charge of Construction and/or Caretaker Site Office representative, and at least two weeks advance notification will be provided prior to the initiation of field work.

Field personnel will be required to have read this Work Plan and the site-specific APP/SSHP prior to starting field work. In addition, prior to mobilization, ARCADIS will host a subcontractor kickoff meeting to discuss communication and coordination logistics; project goals

SAP Worksheet #14 – continued

and quality standards; and safety requirements, protocols and procedures. Daily health and safety tailgate meetings will be conducted with all on-site personnel for the duration of field work.

14.3 Soil Sample Collection

The RI field work will include the collection of soil samples at various depths from 17 soil borings advanced in DRMO South and 12 soil borings advanced in CTA North (Figures 3 and 4, respectively).

Soil borings will be advanced using a truck-mounted, direct-push (Geoprobe®) drill rig. Direct push rods will be driven in four-foot segments by a hydraulic hammer until the specified sample depth is reached. Borings will be continuously cored from the ground surface to depth using a stainless steel dual-tube corer lined with an acetate sleeve. As the core barrel is advanced, soil will be driven into the inner acetate sleeve. After being driven four feet, the rods will be removed from the borehole, and the acetate sleeve containing the soil removed from the sample barrel. The soil will be removed from the sleeve, its characteristics will be logged, and a sample will be collected from the desired depth interval. Lithologic descriptions will be recorded on standard boring log forms by an ARCADIS field geologist using the Unified Soil Classification System. A copy of the boring log form is provided in Attachment 1 and will include descriptions of texture (e.g., relative angularity, roundness, sorting of the particles, grain size), moisture content (e.g., dry, moist, wet, or saturated), color (e.g., referencing Munsell color charts), stratification, depth to water when encountered, sample recovery depths, and other observations (presence of odors, vapors, free or residual petroleum product). Visual observations (e.g., for odors or staining, evidence of greensand) and photoionization detector (PID) screening of the soil column will be used to identify the possible presence of COPCs.

A dual-tube hand auger may be substituted for the Geoprobe if access is limited or if the substitution is required by the biological monitor (e.g., to protect nesting birds). Sample locations requiring the use of a hand auger will be identified during a site walk with the field team lead and biological monitor.

Soil samples will be collected from two or more depth intervals at each boring. Soil samples will be collected from depths of up to 15 feet below ground surface (bgs) at DRMO South and up to 10 feet bgs at CTA North. The depths of the deeper samples (i.e., samples collected from below 0.5 feet bgs) may be biased if visual observations and/or PID screening of the soil column indicate the possible presence of COPCs.

Soil samples will be collected using clean and inert sampling tools and placed in appropriate containers provided by the analytical laboratory. Once labeled, the samples will be packaged in seam-sealing plastic bags and placed in a cooler with ice for transportation to the analytical laboratory, following standard chain-of-custody procedures.

Following sample collection, each borehole will be properly abandoned by backfilling with bentonite grout starting from the bottom of the hole to the ground surface.

SAP Worksheet #14 – continued

Direct Push

The following is a detailed description of the soil sampling procedures via direct push.

1. Advance the borehole until the top of the desired sampling interval is reached.
2. Hydraulically advance a decontaminated 48-inch-long by 2-inch-diameter sampler fitted with an acetate liner into the undisturbed formation.
3. Following sample acquisition, bring sampler to the surface and remove from the drive rod. Loosen the upper and lower fittings (tip and shoe) and take the sampler to the sample handling area.
4. At the sample handling area, remove the fittings and immediately remove the acetate liner from the sampler. Generally, the middle liner is considered the least disturbed and shall be retained as the analytical laboratory sample.
5. The geologist or sample technician will cut the acetate liner (approximately 6-inch section) that represents the sample interval of interest.

Hand Auger

The following is a detailed description of the soil sampling procedures via hand auger.

1. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). Before advancing the auger, it may be advisable to remove the first 3 to 6 inches of surface soil over a radius of approximately 6 inches around the borehole.
2. Attach the auger bit to a drill rod extension, and attach the T-handle to the drill rod.
3. Place the disposable sampler sleeve into the sampler barrel. The sampler barrels are generally 6 inches in length and can hold one 6-inch or two 3-inch sleeves. Assemble the sampler by aligning both sides of the barrel and then attaching the drive shoe and head to the barrel's bottom and top, respectively. Begin augering, periodically removing the sample sleeve.
4. Seal the ends of each sample sleeve with Teflon sheeting and tightly fitting plastic end caps. The end caps shall then be held in place with silicone tape or other USEPA-approved sealing tape. Electrical tape shall not be used.
5. If another sample is to be collected at a greater depth in the same borehole, reattach the auger bit to the drill and assembly and follow the steps above. Decontaminate the auger between samples.
6. After reaching the desired depth, slowly and carefully remove the auger from the borehole.
7. Attach sample label(s) to the container(s).
8. Record required field logbook and sample custody information. Package samples and prepare for transfer or shipment.

SAP Worksheet #14 – continued

9. Destroy the borehole according to applicable state, county, and local regulations.

14.4 Monitoring Well Installation and Sampling

RI field work will include the installation and collection of groundwater samples from temporary groundwater monitoring wells installed at four of the boring locations (DRMOS-SB36, DRMOS-SB37, DRMOS-SB39, and DRMOS-SB42) in DRMO South (Figure 3) and three of the boring locations (CTAN-SB14, CTAN-SB16, and CTAN-SB20) in CTA North (Figure 4).

The temporary wells will be drilled, completed, and developed by a California-licensed drilling subcontractor. The same drill rig will be used for both soil sampling and monitoring well installation in order to minimize the equipment footprint. The drilling subcontractor will work under the oversight of a field geologist who will be responsible for logging the soil column for lithology in accordance with the Unified Soil Classification System. A PID will be utilized to measure VOC levels within the work area breathing zone during drilling as well as when the soil cuttings are visibly affected.

Each well will be completed to an anticipated maximum depth of 15 feet bgs and below the water table, which is expected to be between 6 and 8 feet bgs based on SI field observations. The temporary monitoring wells will be installed using the direct-push (Geoprobe®) dual-tube system, which employs a hydraulically-operated percussion hammer (or similar) to advance an outer steel casing that contains a dual-tube liner for sampling soil. The outer casing isolates shallow layers and permits the unit to continue to probe at depth. When the borehole has been advanced to the desired depth, the monitoring well will be installed through the steel casing.

Monitoring wells will be installed to yield representative samples of the uppermost water-bearing zones (i.e., monitoring wells will be screened across the water table, and well screen length will not exceed 10 feet). The temporary monitoring wells will consist of 2-inch diameter polyvinyl chloride (PVC) slotted screen (0.010-inch) and blank riser. The screen interval will be 10 feet long with approximately 2 feet extending above the water table and 8 feet extending below the water table. Final well and well screen depths will be assessed in the field based on observations of the soil core (e.g., moisture content).

A filter pack consisting of graded silica sand (#3 sand filter pack) will be placed in the annular space as the outer rod is slowly removed, and the well casing will be surged using a vented surge block to facilitate maximum settling of the filter pack. The filter pack will extend approximately 1 to 2 feet above the top of the well screen. Alternatively, the filter pack will be installed as a pre-pack that surrounds the well screen and is emplaced with the PVC screen and riser pipe materials. Pre-pack filters will consist of an outer stainless steel mesh screen that will contain the filter pack (graded silica sand) and hold it against the inner PVC slotted screen. Once the pre-pack well assembly has been lowered, the rod will be retracted to a point above the screen and a sand barrier will be created (e.g., through natural formation collapse or by gravity installation of sand through the rod annulus). It is anticipated that a 2-inch (inner diameter) pre-pack well assembly can be installed in a 4-inch diameter borehole.

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A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the filter pack. Annular space remaining above the bentonite seal will be filled with a bentonite/cement grout mixture to the ground surface. Since the temporary monitoring wells will be abandoned once the field effort is finished, no surface completions will be necessary. A permanent mark (e.g., a notch) will be placed on top of the north side of the well casing to mark the point for water level measurements.

The newly installed wells will be developed to remove formation and filter pack materials from the wells, promote hydraulic communication with the surrounding water-bearing zone, and ensure the collection of representative groundwater samples. The bentonite/cement grout will be allowed to cure for a minimum of 24 hours, after which the wells will be developed by purging with a submersible pump or bailer until approximately three to five well casing volumes have been removed. During this process, indicator parameters (e.g., pH, temperature, and specific conductance) will be measured periodically and recorded along with observations concerning the quantity and clarity of the water withdrawn. The development will be considered complete once the indicator parameters have stabilized and the extracted water appears clear. If poor recharge rates are observed, the monitoring wells will be developed by continuously surging the well for 15 minutes and then bailing the well dry. All purge water generated during the well development activities will be containerized and disposed of in accordance with the Waste Management Plan.

After development, the wells will be allowed to recover for a minimum of 72 hours before sampling. Before each monitoring well is sampled, organic vapor concentrations will be measured at the top of the well using a PID and recorded on the groundwater sampling sheets. Field staff will also measure the groundwater level at each monitoring well using an electronic oil-water interface probe. If the probe indicates that separate phase product is present in the well, the thickness of the product will be measured and recorded on the groundwater sampling sheet. Depth to groundwater and, if applicable, separate phase product (to the nearest 0.01 foot) will be measured from the top of PVC casing (at the permanent mark/notch placed on the north side), and distance from ground surface to top of PVC casing will be measured and recorded in the boring log and field logbook. Separate phase product observed in the well will be removed to the extent possible during purging activities and prior to sample collection, and the groundwater sample will be collected from the approximate midpoint of the water column. If separate phase product cannot be removed during purging activities, a groundwater sample will not be collected.

Disposable nitrile gloves will be worn during all purging and sampling activities. Nitrile gloves will be disposed of after the well is purged, and a new pair will be worn before each well is sampled to avoid possible cross-contamination.

A decontaminated, portable bladder pump will be installed prior to purging and sampling activities. The portable pump will be lowered until the pump inlet reaches the water column midpoint. The pump inlet measurement will be recorded on the Well Sampling Log (Attachment 1).

SAP Worksheet #14 – continued

The water quality meter will be calibrated to the manufacturer's specifications using current (unexpired) standards. Calibration results will be documented in the field logbook. A flow-through cell will be used to monitor the water quality parameters.

Groundwater samples will be collected from the monitoring wells with the portable bladder pump using low-flow methodology in accordance with the 1996 USEPA guidance document Low-Flow (Minimal Drawdown) Groundwater Sampling Procedures (USEPA 1996):

1. Measure the depth to groundwater from the inner PVC casing reference point to the nearest 0.01 foot.
2. Record measurement on the Well Sampling Log to the nearest 0.01 foot.
3. Lower the portable bladder pump.
4. Attach the flow-through cell tubing and pressure hose to the sampling cap.
5. Begin pumping the well at 0.1 to 0.5 liters per minute (100 to 500 milliliters). Check the water level in the well, and measure the discharge rate of the pump by using a graduated cylinder every minute for the first 5 minutes. Ideally, the pumping rate should equal the well recharge rate with little or no water level drawdown in the well. (Drawdown may exceed 0.3 foot during purging but should be stabilized for three consecutive readings prior to sample collection.)
6. Measure and record the water level, discharge rate, and water quality indicator parameters in the well on the Well Sampling Log every 5 minutes during purging. During well purging and stabilization, the waste water will be visually screened for a sheen or product layer. Identification of either will be noted in the groundwater sampling log.
7. Check the discharge tubing for air bubbles during the purging process. If bubbles are visible, pluck the tubing where the bubble is to enable the bubble to pass through the tubing and into the flow-through cell.
8. During purging, monitor pH, temperature, turbidity, specific conductance, oxidation/reduction potential, and dissolved oxygen approximately every 5 minutes with a calibrated water quality meter.
9. The groundwater should be purged until indicator parameters have stabilized. The well will be considered stabilized and ready for sample collection when the indicator parameters have stabilized for three consecutive readings, as follows:
   - Consecutive reading within ± 0.01 foot for water level measurements
   - Consecutive readings within ± 0.1 standard units for pH
   - Consecutive readings within ± 3 percent degree Celsius (ºC) for temperature
   - Consecutive readings within ± 10 percent for turbidity (when turbidity is greater than 10 NTUs, or nephelometric turbidity units)

SAP Worksheet #14 – continued

- Consecutive readings within ± 3 percent microSiemens per centimeter for specific conductance
- Consecutive readings within ± 10 millivolts for oxidation-reduction potential
- Consecutive readings within ± 10 percent for dissolved oxygen (mg/L)

If stabilization is not occurring and the procedure has been strictly followed, then sample collection can take place after one hour of purging or once three (minimum) to six (maximum) casing volumes have been removed. Purging shall also be considered complete when a minimum of three to five well volumes have been removed from the well. A well in which the static water level is slow to recover shall be pumped dry and a sample collected as soon as the water volume in the well has recovered sufficiently to meet sample volume requirements on a parameter by parameter basis, regardless of the volume of water purged. Record the specific information on during purging in the field logbook or on the groundwater sampling log.

1. Once the water quality parameters have stabilized, disconnect the flow-through cell from the pump discharge tubing.
2. Collect samples for analysis of organic compounds, inorganic parameters, and then general chemistry parameters (as applicable). Groundwater samples should be filtered in the field prior to being collected in the appropriate containers.
3. Do not allow containers with preservative to overflow, as overfilling may result in loss of preservative. If a container containing preservative overflows, then discard the sample and resample using a new preserved sample container.
4. Containers should be kept capped, except when they are being filled.
5. Affix a completed sample label to the sample container and cover with clear packing tape. Section 27.2 of this SAP lists the required sample label information.
6. Wrap glass bottles in bubble-wrap packaging material, place into resealable bags, and place sample containers into a cooler containing double-bagged ice. Note: Double-bagged ice should prevent the water generated from melting ice to contact the sample container(s).
7. Record sample number, time and date, and requested analysis on COC form.

The Navy does not anticipate separate phase product will be present in the groundwater. However, during well purging and stabilization, the waste water will be visually screened for a sheen or product layer. Identification of either will be noted in the groundwater sampling log.

14.5 Temporary Well Abandonment

Upon completion of the sampling effort and with concurrence of the Navy, temporary monitoring wells will be abandoned in accordance with Solano County Department of Resource Management guidelines. The sampling system (i.e., PVC casing) will be removed and bentonite chips will be added via tremie pipe to each borehole and hydrated with tap water. The hydration

will occur in 2-foot intervals to ensure hydration across the boring. The grout will be emplaced starting from the bottom of the hole and to the ground surface.

14.6 Land Survey

Each new monitoring well and soil boring location will be surveyed by a California-licensed land surveyor to document the horizontal positions and elevations of each location. Coordinates for each surveyed location will be established to an accuracy of 0.1 feet horizontally and 0.01 feet vertically. All horizontal coordinates will be based on California State Plan Coordinate System, North American Datum 1983, and all vertical coordinates will be based on North American Vertical Datum 1988. Top-of-casing elevations for the temporary monitoring wells will be measured at the mark/notch indicating the top of the north side of the well casing where water level measurements are taken.

14.7 Decontamination Procedures

To prevent cross-contamination, reusable equipment will be decontaminated prior to subsequent use at the site. The following summarizes the decontamination procedures that will be followed during the field investigation:

- All heavy equipment (e.g., down-hole drilling equipment used for the collection of soil samples) will be steam-cleaned on site in a portable unit provided by the contracted drilling company. The equipment will be cleaned until all visible soil and debris is removed.
- Soil collection and groundwater collection/purging equipment, including the associated tooling, will be cleaned after each use by following a three-stage decontamination procedure consisting of a non-phosphate detergent solution (e.g., Alconox or equivalent), a potable water rinse, and a final deionized water rinse. The sample collection equipment will be cleaned until all visible soil and debris is removed.

For equipment rinsate samples, water falling off the sampling equipment from the final rinse will be collected in appropriate sample bottles and analyzed for the same parameters as the field samples. Equipment rinsate blanks will be collected at a rate of one per day for all reusable sampling equipment.

Dedicated tubing will be used for groundwater sample collection.

14.8 Investigation-Derived Waste

All investigation-derived waste (i.e., excess soil from soil coring activities and soil cuttings, water from decontamination activities, purge water, etc.) will be stored in appropriately labeled, Department of Transportation-approved 55-gallon drums staged in a selected area of the Site. Open topped drums will be closed and secured with the bolt at the end of each day and when full. A representative waste sample will be collected from the waste stored in each container using a

clean stainless steel trowel for soil or a clean disposable bailer for liquid waste. For soil waste, a composite sample will be collected from several areas within each drum. The waste characterization samples will be transferred into clean, laboratory-provided containers and placed in a cooler with ice for transportation to the analytical laboratory under chain-of-custody protocol. Waste characterization samples will be analyzed for the compounds required by the disposal facility. Further details of the waste handling and sampling procedures are provided in the Waste Management Plan.

Following waste characterization and profiling, the containers will be transferred to the waste disposal contractor for final offsite disposal at a disposal facility certified to accept Resource Conservation and Recovery Act (RCRA) or non-RCRA hazardous waste, as appropriate. The appropriate disposal facility will be determined based on the following hazardous classifications.

- Although not anticipated to be present, any soil or water classified as Resource Conservation and Recovery Act (RCRA) hazardous waste will be transported to a licensed treatment, storage, and disposal facility.
- Any soil or water classified as non-RCRA hazardous waste will be transported to a licensed hazardous waste landfill facility.
- Soil classified as nonhazardous or inert waste will be transported to a licensed landfill or soil treatment facility permitted to accept such waste. Water classified as nonhazardous will be transported to a licensed disposal facility.

A Generator Waste Profile form will be prepared and must be approved by the selected disposal facility prior to shipment. ARCADIS will prepare the profile(s) and the appropriate hazardous/non-hazardous waste manifests that will accompany each shipment for signature by the designated Navy representative(s). Two photocopies will be made of each manifest signed by the transporter; one copy will be provided to the Navy and one copy will be kept in the project file. All wastes will be transported via a licensed transporter.

14.9 Data Management and Review

The following sections describe how both field and laboratory data will be managed during this investigation.

Field Logbooks

A permanently bound field logbook with consecutively numbered pages, used for sampling activities only, will be assigned to this project. All entries will be recorded in indelible ink. The logbook pages will be signed by the responsible sampler at the end of each workday, and any unused portions of the logbook pages will be crossed out, signed, and dated.

If it is necessary to transfer the logbook to another person, the person relinquishing the logbook will sign and date the last page used, and the person receiving the logbook will sign and date the next page to be used.

SAP Worksheet #14 – continued

At a minimum, the logbook will contain the following information:

- Project name and location
- Date and time
- Personnel in attendance
- General weather information
- Work performed
- Field observations
- Sampling performed, including specific information such as location, type of sample, type of analysis, and sample identification
- Field analysis performed, including results, instrument checks, problems, and calibration records for field instruments
- Descriptions of deviations from this SAP
- Problems encountered and corrective action taken
- Identification of field QC samples
- Verbal or written instructions
- Any other events that may affect the samples.

Variances or changes to field activities will be documented in the field logbook.

Document Corrections

Any project documentation will be changed or corrected by crossing out the erroneous item 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 cross-out line. The new information should be written clearly above the crossed-out item.

Laboratory Data Management

The analytical data validation component follows NAVFAC SW Environmental Work Instruction (EWI) #1). The analytical data (with the exception of waste characterization data) will be sent to an independent, third-party data validation company. Ten (10) percent of the data will be subject to a USEPA Level IV validation, and the remaining 90 percent of the data will undergo USEPA Level III validation. The 10 percent portion of the data set will be comprised of routine field samples and field QC samples (e.g., field duplicates, field blanks, trip blanks, and equipment rinsate blanks). The validator will perform calculation checks for these data and the data for the associated QC samples (e.g., matrix spike/matrix spike duplicate, laboratory control sample, etc.). The chemical data will be validated and qualified per EWI #1 (Southwest Division Naval Facilities Engineering Command [SWDIV] 2001), USEPA National Functional

SAP Worksheet #14 – continued

Guidelines for Inorganic Superfund Data Review (2010), USEPA National Functional Guidelines for Organic Methods Data Review (2008), as applicable.

The flagging associated with the third-party data validation report will be entered into the database for each sample and used during data interpretation activities. The analytical data will be uploaded to the Naval Installation Restoration Information Solution (NIRIS) database in the Naval Electronic Data Deliverable format per EWI #6 (SWDIV 2005).

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SAP Worksheet #15 — Reference Limits and Evaluation Tables

15.1 – Groundwater

Matrix: Groundwater
Analytical Group: TPH-e (8015B)

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (µg/L)	Laboratory Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Diesel (C10-C28)	68334-30-5	100	Water Board ESL	50	50	20	15.9
Motor Oil (C28-C40)	-35282	100	Water Board ESL	300	300	100	84

Notes:
1 Project action limits are the 2013 groundwater environmental screening levels (ESLs) where groundwater is a current or potential drinking water resource (Table F-1a; Water Board 2013).
2 Because a CAS number does not exist for this compound, the NIRIS code was used.

µg/L	micrograms per liter
CAS	Chemical Abstract Service
DL	detection limit
ESL	Environmental Screening Level
LOD	limit of detection
LOQ	limit of quantitation
Water Board	Regional Water Quality Control Board
TPH-e	total petroleum hydrocarbons - extractable

SAP Worksheet #15.1 – continued

Matrix: Groundwater
Analytical Group: PAH (8270C-SIM)

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (µg/L)	Laboratory Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
2-Methylnaphthalene	91-57-6	27	USEPA RSL	0.1	0.1	0.04	0.03
Acenaphthene	83-32-9	400	USEPA RSL	0.1	0.1	0.04	0.0202
Acenaphthylene	208-96-8	0.42[2]	USEPA RSL	0.1	0.1	0.04	0.02
Anthracene	120-12-7	1,300	USEPA RSL	0.1	0.1	0.04	0.031
Benzo(a)anthracene	56-55-3	0.029[4]	USEPA RSL	0.1	0.1	0.04	0.02
Benzo(a)pyrene	50-32-8	0.0029[4]	USEPA RSL	0.1	0.1	0.04	0.02
Benzo(b)fluoranthene	205-99-2	0.029[4]	USEPA RSL	0.1	0.1	0.04	0.02
Benzo(k)fluoranthene	207-08-9	0.29	USEPA RSL	0.1	0.1	0.04	0.02
Benzo(g,h,i)perylene	191-24-2	0.10	Water Board ESL	0.1	0.1	0.04	0.0253
Chrysene	218-01-9	2.9	USEPA RSL	0.1	0.1	0.04	0.0273
Dibenzo(a,h)anthracene	53-70-3	0.0029[4]	USEPA RSL	0.1	0.1	0.04	0.02
Fluoranthene	206-44-0	630	USEPA RSL	0.1	0.1	0.04	0.02
Fluorene	86-73-7	220	USEPA RSL	0.1	0.1	0.04	0.02
Indeno(1,2,3-cd)pyrene	193-39-5	0.029[4]	USEPA RSL	0.1	0.1	0.04	0.02
Naphthalene	91-20-3	0.14	USEPA RSL	0.1	0.1	0.04	0.0222
Phenanthrene	85-01-8	26[3]	USEPA RSL	0.1	0.1	0.04	0.02
Pyrene	129-00-0	87	USEPA RSL	0.1	0.1	0.04	0.024

Notes:

1 Project action limits are the 2013 USEPA residential tap water regional screening levels (RSLs) or the 2013 groundwater environmental screening levels (ESLs) where groundwater is a potential source of drinking water (Table F-1a; Water Board 2013). For chemicals that lack toxicity criteria, surrogate chemicals were chosen based on similar chemical structures.

2 1,4-Dichlorobenzene (CAS No. 106-46-7) used as a surrogate.

3 2-Phenylphenol (CAS No. 90-43-7) used as a surrogate.

4 The Quantitation Limit Goal (Laboratory LOQ) exceeds the project action limit (USEPA RSL). If Laboratory LOQs for actual samples are greater than the project action limits listed in this table, the laboratory will report data to the LOD and decisions will be based on the Laboratory LOD. Note that ingestion of groundwater is not anticipated to be considered a complete exposure pathway for human health.

µg/L micrograms per liter
CAS Chemical Abstract Service
DL detection limit
LOD limit of detection
LOQ limit of quantitation
QL quantitation limit
RSL Regional Screening Level
SIM selected ion measurement
USEPA United States Environmental Protection Agency

SAP Worksheet #15.1 – continued

Matrix: Groundwater
Analytical Group: Metals/Mercury (6020/7470)

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (µg/L)	Laboratory Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Aluminum	7429-90-5	480	Background	50	50	25	10.8
Antimony	7440-36-0	5.6	Background	1	1	0.5	0.144
Arsenic	7440-38-2	78	Background	1	1	1	0.257
Barium	7440-39-3	1,200	Background	1	1	0.5	0.253
Beryllium	7440-41-7	16	USEPA RSL	1	1	0.25	0.201
Cadmium	7440-43-9	16	Background	1	1	0.5	0.129
Chromium	7440-47-3	22	Background	1	1	0.25	0.228
Cobalt	7440-48-4	100	Background	1	1	0.25	0.233
Copper	7440-50-8	33	Background	1	1	1	0.526
Iron	7439-89-6	11,000	USEPA RSL	50	50	25	7.22
Lead	7439-92-1	10	Background	1	1	0.5	0.291
Manganese	7439-96-5	5400	Background	1	1	0.5	0.175
Mercury	7439-97-6	0.63	USEPA RSL	0.2	0.2	0.2	0.0361
Molybdenum	7439-98-7	8.8	Background	1	1	0.5	0.245
Nickel	7440-02-0	7.5	Background	1	1	0.5	0.177
Selenium	7782-49-2	12	Background	1	1	0.5	0.258
Silver	7440-22-4	71	USEPA RSL	1	1	0.25	0.139
Thallium	7440-28-0	2	USEPA MCL	1	1	0.25	0.161
Vanadium	7440-62-2	140	Background	1	1	0.5	0.273
Zinc	7440-66-6	260	Background	5	5	5	3.01

Notes:

1 Project action limits are background/ambient concentrations taken from the Final Compilation of Technical Memoranda on Ambient Analyses of Metals in Soils and Groundwater, Mare Island, California (Tetra Tech EM, Inc. [TtEMI] 2002), if available, or 2013 USEPA residential tap water RSLs or 2009 USEPA MCLs.

µg/L micrograms per liter
CAS Chemical Abstract Service
DL detection limit
LOD limit of detection
LOQ limit of quantitation
MCL maximum contaminant level
RSL Regional screening level
USEPA United States Environmental Protection Agency

SAP Worksheet #15.1 – continued

Matrix: Groundwater
Analytical Group: PCBs (8082)

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (ug/L)	Laboratory Specific Limit		
					LOQ (ug/L)	LOD (ug/L)	DL (ug/L)
Aroclor-1016	12674-11-2	0.96	USEPA RSL	0.5	0.5	0.2	0.16
Aroclor-1221	11104-28-2	0.0040[2]	USEPA RSL	1	1	0.6	0.324
Aroclor-1232	11141-16-5	0.0040[2]	USEPA RSL	0.5	0.5	0.2	0.142
Aroclor-1242	53469-21-9	0.034[2]	USEPA RSL	0.5	0.5	0.2	0.159
Aroclor-1248	12672-29-6	0.034[2]	USEPA RSL	0.5	0.5	0.2	0.16
Aroclor-1254	11097-69-1	0.034[2]	USEPA RSL	0.5	0.5	0.2	0.157
Aroclor-1260	11096-82-5	0.034[2]	USEPA RSL	0.5	0.5	0.2	0.134

Notes:

1 Project action limits are the 2013 USEPA residential tap water regional screening levels (RSLs).

2 The Quantitation Limit Goal (Laboratory LOQ) exceeds the project action limit (USEPA RSL). If Laboratory LOQs for actual samples are greater than the project action limits listed in this table, the laboratory will report data to the LOD and decisions will be based on the Laboratory LOD. Note that ingestion of groundwater is not anticipated to be considered a complete exposure pathway for human health.

µg/L micrograms per liter
CAS Chemical Abstract Service
DL detection limit
LOD limit of detection
LOQ limit of quantitation
PCB polychlorinated biphenyl
RSL Regional Screening Level
USEPA United States Environmental Protection Agency

15.2 – Soil

Matrix: Soil
Analytical Group: TPH-e (8015B)

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory Specific Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Diesel (C10-C28)	68334-30-5	100	Water Board ESL	1	1	0.4	0.278
Motor Oil (C28-C40)	-35282	500	Water Board ESL	5	5	1.6	1.2

Notes:
1 Project action limits are the 2013 shallow soil (less than 3 meters below ground surface) environmental screening levels (ESLs) for residential use where groundwater is not a source of drinking water (Table B; Water Board 2013).
2 Because a CAS number does not exist for this compound, the NIRIS code was used.

CAS	Chemical Abstract Service
DL	detection limit
ESL	Environmental Screening Level
LOD	limit of detection
LOQ	limit of quantitation
mg/kg	milligrams per kilogram
Water Board	Regional Water Quality Control Board
TPH-e	total petroleum hydrocarbon - extractable

SAP Worksheet #15.2 – continued

Matrix: Soil
Analytical Group: PAHs (8270C-SIM)

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (µg/kg)	Laboratory Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
2-Methylnaphthalene	91-57-6	230,000	Water Board, Table K-1	5	5	2	1
Acenaphthene	83-32-9	3,400,000	Water Board, Table K-1	5	5	2	1
Acenaphthylene	208-96-8	13,000	Water Board ESL2	5	5	2	1
Anthracene	120-12-7	23,000,000	Water Board, Table K-1	5	5	2	1
Benzo(a)anthracene	56-55-3	380	Water Board, Table K-1	5	5	2	1
Benzo(a)pyrene	50-32-8	920	Background3	5	5	2	1
Benzo(b)fluoranthene	205-99-2	380	Water Board, Table K-1	5	5	2	1
Benzo(k)fluoranthene	207-08-9	380	Water Board, Table K-1	5	5	2	1
Benzo(g,h,i)perylene	191-24-2	27,000	Water Board ESL2	5	5	2	1.23
Chrysene	218-01-9	3,800	Water Board, Table K-1	5	5	2	1
Dibenzo(a,h)anthracene	53-70-3	110	Water Board, Table K-1	5	5	2	1
Fluoranthene	206-44-0	2,300,000	Water Board, Table K-1	5	5	2	1
Fluorene	86-73-7	3,100,000	Water Board, Table K-1	5	5	2	1
Indeno(1,2,3-cd)pyrene	193-39-5	380	Water Board, Table K-1	5	5	2	1.01
Naphthalene	91-20-3	1,700	Water Board, Table K-1	5	5	2	1
Phenanthrene	85-01-8	11,000	Water Board ESL2	5	5	2	1
Pyrene	129-00-0	3,400,000	Water Board, Table K-1	5	5	2	1

Notes:

1 Project action limits are the 2013 human health direct exposure soil screening levels for the residential exposure scenario (Table K-1; Water Board 2013) unless otherwise indicated.

2 Project action limits are the 2013 shallow soil (less than 3 meters below ground surface) environmental screening levels (ESLs) for residential use where groundwater is not a source of drinking water (Table B; Water Board 2013).

SAP Worksheet #15.2 – continued

3 Project action limit is the 95th percentile background value taken from the Background Levels of Polycyclic Aromatic Hydrocarbons in Northern California Surface Soil (PG&E and Navy 2002).

µg/kg	micrograms per kilogram
CAS	Chemical Abstract Service
DL	Detection Limit
LOD	limit of detection
LOQ	limit of quantitation
PAH	polycyclic aromatic hydrocarbon
Water Board	Regional Water Quality Control Board
SIM	selected ion monitoring

SAP Worksheet #15.2 – continued

Matrix: Soil
Analytical Group: Metals/Mercury (6010/7471)

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory Specific Limit		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aluminum	7429-90-5	35,000	Background	5	5	2.5	1.31
Antimony	7440-36-0	8.5	Background	0.5	0.5	0.5	0.159
Arsenic	7440-38-2	36	Background	0.25	0.25	0.125	0.083
Barium	7440-39-3	15,000	Water Board, Table K-1	0.25	0.25	0.0625	0.0537
Beryllium	7440-41-7	0.9	Background	0.1	0.1	0.025	0.019
Cadmium	7440-43-9	5.2	Background	0.25	0.25	0.0313	0.0255
Chromium	7440-47-3	140	Background	0.25	0.25	0.125	0.063
Cobalt	7440-48-4	0.33	Water Board, Table K-1	0.25	0.25	0.0625	0.0299
Copper	7440-50-8	120	Background	0.259	0.259	0.25	0.0864
Iron	7439-89-6	62,000	Background	5	5	5	1.63
Lead	7439-92-1	59	Background	0.25	0.25	0.25	0.073
Manganese	7439-96-5	1,600	Background	0.25	0.25	0.25	0.0512
Mercury	7439-97-6	2	Background	0.02	0.02	0.01	0.00629
Molybdenum	7439-98-7	390	Water Board, Table K-1	0.25	0.25	0.25	0.0561
Nickel	7440-02-0	130	Background	0.25	0.25	0.25	0.0677
Selenium	7782-49-2	390	Water Board, Table K-1	0.5	0.5	0.5	0.16
Silver	7440-22-4	390	Water Board, Table K-1	0.25	0.25	0.125	0.0748
Thallium	7440-28-0	0.78	Water Board, Table K-1	25	0.5	0.5	0.163
Vanadium	7440-62-2	190	Background	0.5	0.25	0.25	0.057
Zinc	7440-66-6	230	Background	0.25	1	0.5	0.0975

Notes:

1 Project action limits are background/ambient concentrations taken from the Final Compilation of Technical Memoranda on Ambient Analyses of Metals in Soils and Groundwater, Mare Island, California (TtEM 2002), if available, or 2013 human health direct exposure soil screening levels for the residential exposure scenario (Table K-1; Water Board 2013).

CAS Chemical Abstract Service
DL detection limit
LOD limit of detection
LOQ limit of quantitation
mg/kg milligrams per kilogram
Water Board Regional Water Quality Control Board

SAP Worksheet #15.2 – continued

Matrix: Soil
Analytical Group: PCBs (8082)

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory Specific Limit		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aroclor-1016	12674-11-2	3.9	USEPA RSL	0.012	0.012	0.003	0.00296
Aroclor-1221	11104-28-2	0.14	USEPA RSL	0.024	0.024	0.009	0.00796
Aroclor-1232	11141-16-5	0.14	USEPA RSL	0.012	0.012	0.006	0.00371
Aroclor-1242	53469-21-9	0.22	USEPA RSL	0.012	0.012	0.006	0.00284
Aroclor-1248	12672-29-6	0.22	USEPA RSL	0.012	0.012	0.003	0.00214
Aroclor-1254	11097-69-1	0.22	USEPA RSL	0.012	0.012	0.006	0.00305
Aroclor-1260	11096-82-5	0.22	USEPA RSL	0.012	0.012	0.003	0.00113

Notes:
1 Project action limits are the 2013 USEPA residential soil regional screening levels (RSLs).

CAS Chemical Abstract Service
DL detection limit
LOD limit of detection
LOQ limit of quantitation
mg/kg milligrams per kilogram
PCB polychlorinated biphenyl
RSL Regional Screening Level
USEPA United States Environmental Protection Agency

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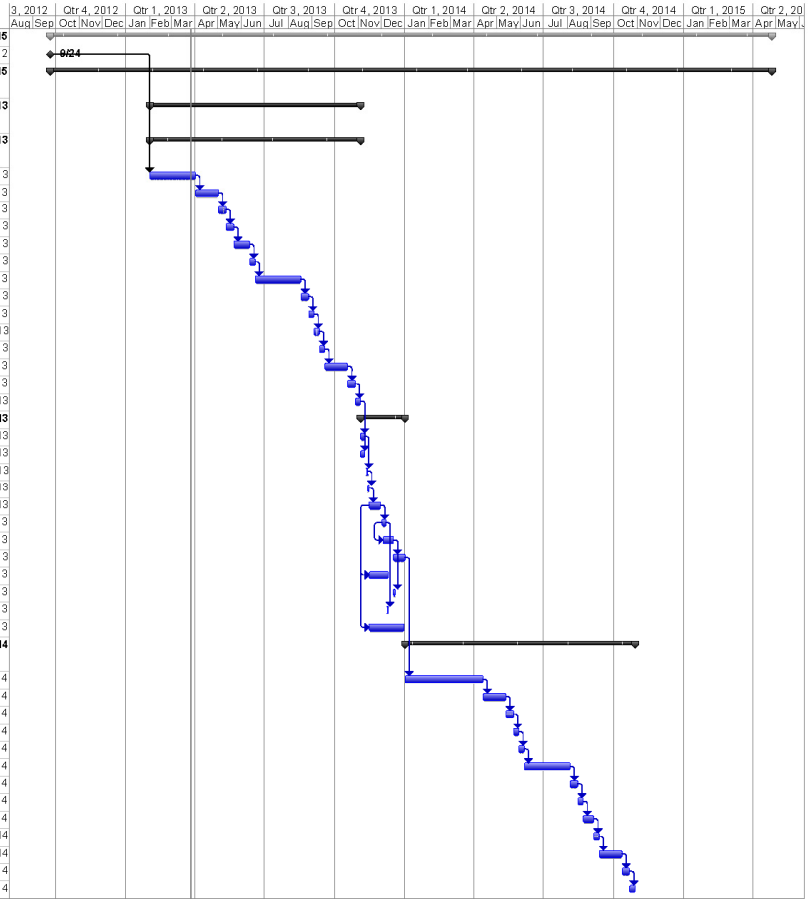
SAP Worksheet #16 — Project Schedule/Timeline Table

Schedule PTO X001, Remedial Investigation for Crane Test Area North and Defense Reutilization and Marketing Office South
Former Mare Island Naval Shipyard, Vallejo, California

ID	Task Name	Duration	Start	Finish
0	**Mare Island - Remedial Investigation**	**655 days**	**Mon 9/24/12**	**Sun 4/26/15**
1	Contract Award	0 edays	Mon 9/24/12	Mon 9/24/12
2	**Work Element No. 1 - Project Management, Meetings, Administrative Support**	**655 days**	**Mon 9/24/12**	**Sun 4/26/15**
5	**Work Element No. 2 - Remedial Investigation (RI) Work Plan**	**191 days**	**Fri 2/1/13**	**Mon 11/4/13**
6	**Remedial Action Work Plan (including all supporting plans)**	**191 days**	**Fri 2/1/13**	**Mon 11/4/13**
7	Internal Draft RI Work Plan (WP/SSHP/SAP)	60 edays	Fri 2/1/13	Tue 4/2/13
8	Navy Review	30 edays	Tue 4/2/13	Thu 5/2/13
9	Response to Navy RPM/ROICC Comments	10 edays	Thu 5/2/13	Sun 5/12/13
10	Revised Internal Draft SAP QAO Comments	10 edays	Sun 5/12/13	Wed 5/22/13
11	Navy/QAO Review and Comment Resolution	21 edays	Wed 5/22/13	Wed 6/12/13
12	Draft RI Work Plan (WP/SSHP/SAP)	7 edays	Wed 6/12/13	Wed 6/19/13
13	Regulatory Review	60 edays	Wed 6/19/13	Sun 8/18/13
14	Response to Regulatory Comments	10 edays	Sun 8/18/13	Wed 8/28/13
15	Internal Draft Final RI Work Plan (WP/SSHP/SAP)	7 edays	Wed 8/28/13	Wed 9/4/13
16	Navy/QAO Review and Approval	7 edays	Wed 9/4/13	Wed 9/11/13
17	Draft Final RI Work Plan (WP/SSHP/SAP)	7 edays	Wed 9/11/13	Wed 9/18/13
18	Regulatory Review	30 edays	Wed 9/18/13	Fri 10/18/13
19	Response to Regulatory Comments	10 edays	Fri 10/18/13	Mon 10/28/13
20	Final RI Work Plan (WP/SSHP/SAP)	7 edays	Mon 10/28/13	Mon 11/4/13
21	**Work Element No. 3 - Field Work**	**39 days**	**Mon 11/4/13**	**Tue 12/31/13**
22	Pre-field Activities	5 days	Mon 11/4/13	Fri 11/8/13
23	Permitting	5 days	Mon 11/4/13	Fri 11/8/13
24	Mobilization and Site Preparation	1 day	Tue 11/12/13	Tue 11/12/13
25	Utility Clearance	2 days	Wed 11/13/13	Thu 11/14/13
26	Soil Sampling and Well Installation	10 days	Fri 11/15/13	Fri 11/29/13
27	Groundwater Sampling	5 days	Mon 12/2/13	Fri 12/6/13
28	Laboratory Analysis	10 days	Tue 12/3/13	Mon 12/16/13
29	Data Validation	10 days	Tue 12/17/13	Tue 12/31/13
30	Biological Monitoring and Avoidance	16 days	Fri 11/15/13	Mon 12/9/13
31	Land Survey	2 days	Tue 12/17/13	Wed 12/18/13
32	Site Restoration and Demobilization	1 day	Mon 12/9/13	Mon 12/9/13
33	IDW Management, Profiling and Disposal	30 days	Fri 11/15/13	Mon 12/30/13
34	**Work Elements No. 4 - Remedial Investigation Reports**	**212 days**	**Tue 12/31/13**	**Tue 10/28/14**
35	Internal Draft RI Reports	102 edays	Tue 12/31/13	Sat 4/12/14
36	Navy Review	30 edays	Sat 4/12/14	Mon 5/12/14
37	Response to Navy Comments	10 edays	Mon 5/12/14	Thu 5/22/14
38	Navy QAO SAP Review	7 edays	Thu 5/22/14	Thu 5/29/14
39	Draft RI Report	7 edays	Thu 5/29/14	Thu 6/5/14
40	Regulatory Review	60 edays	Thu 6/5/14	Mon 8/4/14
41	Response to Regulatory Comments	10 edays	Mon 8/4/14	Thu 8/14/14
42	Internal Draft Final RI Report	7 edays	Thu 8/14/14	Thu 8/21/14
43	Navy Review and Approval	14 edays	Thu 8/21/14	Thu 9/4/14
44	Draft Final RI Report	7 edays	Thu 9/4/14	Thu 9/11/14
45	Regulatory Review	30 edays	Thu 9/11/14	Sat 10/11/14
46	Response to Regulatory Comments	10 edays	Sat 10/11/14	Tue 10/21/14
47	Final RI Report	7 edays	Tue 10/21/14	Tue 10/28/14



Project: Mare Island - Remedial Investi
Date: Wed 3/27/13
e days = calendar days
days = working days

Task | Split | Milestone | Summary | Project Summary | External Tasks | External Milestone | Inactive Task | Inactive Milestone | Inactive Summary | Manual Task | Duration-only | Manual Summary Rollup | Manual Summary | Start-only | Finish-only | Progress | Deadline

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SAP Worksheet #17 — Sampling Design and Rationale

This section describes the sampling design and rationale in terms of the media that will be sampled and the analytical groups that will be evaluated by the off-site laboratory.

In general, the locations and depths of the proposed samples were chosen based on the preliminary conceptual site model to: (1) confirm and/or further delineate areas of potential chemical of potential concern [COPC] impacts identified through review of the 2012 site inspection [SI] data and historical Investigation Area H2 remedial investigation [RI] data; and (2) fill in data gap areas. A discussion of the SI results is provided in Worksheet 10, Section 10.6.1.

Potential COPCs included those chemicals that were detected at concentrations above the applicable ecological and/or human health screening criteria in soil and/or groundwater at DRMO South or CTA North during the SI. These included:

- Polychlorinated biphenyls (PCBs; specifically Aroclor 1260), total petroleum hydrocarbons as diesel (TPH-d), total petroleum hydrocarbons as motor oil (TPH-mo), polycyclic aromatic hydrocarbons (PAHs), and metals in soil at DRMO South;
- PAHs, metals, pesticides (endrin), and volatile organic compounds (chloroform) in groundwater at DRMO South;
- PCBs (Aroclor 1260), TPH-d, TPH-mo, PAHs, and metals in soil at CTA North; and
- PAHs and metals in groundwater at CTA North.

In general, potential COPCs were selected for further sampling as part of the RI field work if:

- They were considered to be significant risk drivers given anticipated future land use at the sites (light industrial and open space at DRMO South, and open space at CTA North);
- Distribution of the exceedance locations (i.e., sample locations having concentrations exceeding the screening criteria) indicated a potential source that could be further delineated (e.g., 1925-1942 fill material area; COPCs associated with the adjoining remedial action areas at the DRMO and CTA sites; releases from storage tanks, vehicles, or equipment); and
- Additional data were required to assess potential migration pathways or complete a defensible risk risk assessment (e.g., a minimum of six samples is usually recommended for calculating exposure point concentrations for risk assessment purposes).

Samples proposed at DRMO South included:

- Two soil samples collected from one boring location (DRMOS-SB27) near the 2009-2010 Petroleum Corrective Action (PCA) area boundary to further delineate the area of potential TPH-d impacts that is currently defined by historical sample location 02GB026. TPH-d was detected at a concentration of 3,870 milligrams per kilogram (mg/kg; estimated value) in the soil sample collected from 10.5 feet below ground surface (bgs) at 02GB026.

- Four soil samples collected from two boring locations (DRMOS-SB28 and DRMOS-SB29) to delineate the PAH impacts observed at SI soil sample location DRMOS-SB02. One of the two proposed boring locations (DRMOS-SB28) was placed between DRMOS-SB02 and the non-time critical removal action (NTCRA) area boundary to evaluate whether the PAH concentrations observed at DRMOS-SB02 may be associated with PAHs in soil that were targeted during the NTCRA.
- Two soil samples collected from one boring location (DRMOS-SB30) to confirm the isolated area of lead and TPH-d impacts that is currently defined by historical boring location 01GB073. Elevated lead and TPH-d were detected in samples collected from 01GB073 at up to 3 feet bgs.
- Six soil samples collected from three boring locations (DRMOS-SB31 through DRMOS-SB33) to further delineate the TPH (diesel and motor oil) impacts observed at SI soil sample location DRMOS-SB04.
- Two soil samples collected from one boring location (DRMOS-SB34) near the PCA area boundary to further delineate the area of potential TPH-mo impacts that is currently defined by historical sample location DRMOGB046. TPH-mo was detected at a concentration of 5,800 mg/kg in the soil sample collected from 14 feet bgs at DRMOGB046.
- Four soil samples collected from one boring location (DRMOS-SB35) to further delineate the area of TPH (diesel and motor oil) impacts observed in the southeastern portion of the site adjacent to Azuar Drive.
- 20 soil samples and three groundwater samples collected from five boring locations (DRMOS-SB36 through DRMOS-SB40) to: (1) further delineate the area of metals [i.e., lead], Aroclor 1260, and TPH [diesel and/or motor oil] impacts to soil located in the southern portion of the site; and (2) assess the potential for COPC migration from adjoining and hydraulically upgradient areas. DRMO South is adjacent to the Pump Station 1 area and overlaps with Installation Restoration Site 10 (IR10, the Building 831, former PCB storage area) to the south.
- Three soil samples collected from one boring location (DRMOS-SB41) to confirm the isolated area of TPH-d and TPH-mo impacts that is currently defined by historical sample location 10GB050. TPH-d was detected at a concentration of 11,200 mg/kg in a soil sample collected from 5.5 feet bgs at 10GB050; and TPH-mo was detected at an estimated concentration of 1,280 mg/kg in a soil sample collected from 2.5 feet bgs at 10GB050.
- Three soil samples and one groundwater sample collected from one boring location (DRMOS-SB42) to acquire additional data representative of the undeveloped/open space area.
- Two soil samples collected from one boring location (DRMOS-SB43) to confirm the isolated area of metals detections currently defined by historical sample location 01SA001.

Sample locations are shown on Figure 3, and sampling procedures are summarized in Worksheet 14. Limited additional groundwater samples were proposed because more than six groundwater samples (11 total distributed across the site) were collected from DRMO South during the 2012 SI. A minimum of six samples is typically recommended for calculating exposure point concentrations (EPCs) for risk assessment purposes. Furthermore, the groundwater analytical results indicated that: (1) detected constituents were generally below screening criteria; and (2) detections of constituents at elevated concentrations in soil [e.g., PAHs, TPHs, etc.] did not translate to elevated concentrations of these constituents in groundwater. Groundwater samples were proposed at boring locations DRMOS-SB36, DRMOS-SB37, and DRMOS-SB39 to assess potential impacts associated with groundwater migration from hydraulically upgradient source areas (e.g., IR10/Solid Waste Management Unit [SWMU] 107 [the former PCB storage area] and Installation Restoration Site 14 [Pump Station 1 area]). A groundwater sample was proposed at boring location DRMOS-SB42 because groundwater data were not collected from the open space/undeveloped portion of the site during the 2012 SI.

At CTA North, proposed samples included:

- 16 soil samples collected from four boring locations (CTAN-SB14 through CTAN-SB17) to delineate the PAH, metals (primarily lead and arsenic), and TPH impacts observed in shallow soil within the 1925-1942 fill material area (e.g., at CTAN-SB08 and CTAN-SB09). Two groundwater samples will also be collected—one from CTAN-SB14 and one from CTAN-SB16, both located downgradient of CTAN-SB09—to evaluate potential impacts to groundwater in this area.
- 12 soil samples collected from three boring locations (CTAN-SB18 through CTAN-SB20) to delineate the metals (primarily lead and arsenic) and TPH-mo impacts observed in shallow soil within the 1925-1942 fill material area (e.g., at CTAN-SB05 and CTAN-SB06). One groundwater sample will also be collected from CTAN-SB20, located hydraulically downgradient of the CTA removal area footprint, to evaluate potential impacts to groundwater in this area.
- Six soil samples collected from three boring locations (CTAN-SB21 through CTAN-SB23) to further investigate/delineate the isolated area of shallow PAH and TPH impacts that is currently defined by SI sample location CTAN-SB03. CTAN-SB21 and CTAN-SB23 will be located near the electrical utility line that runs through the northeastern portion of CTA North to determine whether the PAH and/or TPH detections at CTAN-SB03 may be associated with the utility corridor fill material. Soil samples collected from these locations will also be visually inspected for the presence of greensand (abrasive blast material).
- Two soil samples collected from one boring location (CTAN-SB24) to further delineate the area of PAH impacts defined by two SI soil sample locations (CTAN-SB12 and CTAN-SB13).Three soil samples collected from one boring location (CTAN-SB25) near the PCA area boundary to confirm the area of potential TPH-d impacts that is currently defined by historical sample location 01GB080. TPH-d was detected at concentrations ranging from 497 mg/kg (estimated value) to 2,640 mg/kg in soil samples collected from 2.5 to 8.5 feet bgs at 01GB080.

Note that three additional groundwater samples are proposed at CTA North because only four groundwater samples were collected from this site during the 2012 SI and these samples were not necessarily collected at or hydraulically downgradient of the most impacted sampling locations (e.g., CTAN-SB09). In addition, a minimum of six samples is usually recommended for calculating EPCs for risk assessment purposes. Sample locations are shown on Figure 4, and soil and groundwater sampling procedures are summarized in Worksheet 14, Sections 14.3 and 14.4, respectively.

Analytical methods were selected based on review of the 2012 SI data and historical IA H2 RI data as well as known COPCs at adjacent sites. Soil samples collected from DRMO South and CTA North will be prepared and analyzed for one or more of the following:

- TPH-extractable (which includes TPH-d and TPH-mo) by United States Environmental Protection Agency (USEPA) Method 3550B/8015B (with silica gel cleanup)
- PAHs by USEPA Method 3550B/8270C-SIM
- Metals by USEPA Method 3050B/6010B
- Mercury by USEPA Method 7471A
- PCBs by USEPA Method 3550B/8082

Groundwater samples collected from DRMO South and CTA North will be prepared and analyzed for one or more of the following:

- TPH-e by USEPA Method 3520C/8015B
- PAHs by USEPA Method 3520C/8270C-SIM
- Metals by USEPA Method 3010A/6020
- Mercury by USEPA Method 7470A
- PCBs by USEPA Method 3520C/8082

SAP Worksheet #18 — Sampling Locations and Methods/SOP Requirements Table

DRMO South

Sampling Location/ID Number	Matrix	Depth[1] (Feet)	Analytical Group	Number of Samples	Sampling SOP Reference
DRMOS-SB27-1	Soil	10 – 11	TPH-e	1	SAP Worksheet #17
DRMOS-SB27-2	Soil	13 – 14	TPH-e	1	SAP Worksheet #17
DRMOS-SB28-1	Soil	0 – 0.5	PAHs	1	SAP Worksheet #17
DRMOS-SB28-2	Soil	1 – 5	PAHs	1	SAP Worksheet #17
DRMOS-SB29-1	Soil	0 – 0.5	PAHs	1	SAP Worksheet #17
DRMOS-SB29-2	Soil	1 – 5	PAHs	1	SAP Worksheet #17
DRMOS-SB30-1	Soil	0 – 0.5	TPH-e, Metals	1	SAP Worksheet #17
DRMOS-SB30-2	Soil	2.5 – 3.5	TPH-e, Metals	1	SAP Worksheet #17
DRMOS-SB31-1	Soil	0 – 0.5	TPH-e	1	SAP Worksheet #17
DRMOS-SB31-2	Soil	1 – 5	TPH-e	1	SAP Worksheet #17
DRMOS-SB32-1	Soil	0 – 0.5	TPH-e	1	SAP Worksheet #17
DRMOS-SB32-2	Soil	1 – 5	TPH-e	1	SAP Worksheet #17
DRMOS-SB33-1	Soil	0 – 0.5	TPH-e	1	SAP Worksheet #17
DRMOS-SB33-2	Soil	1 – 5	TPH-e	1	SAP Worksheet #17
DRMOS-SB34-1	Soil	8 – 13	TPH-e	1	SAP Worksheet #17
DRMOS-SB34-2	Soil	13 – 15	TPH-e	1	SAP Worksheet #17
DRMOS-SB35-1	Soil	0 – 0.5	TPH-e	1	SAP Worksheet #17
DRMOS-SB35-2	Soil	1 – 5	TPH-e	1	SAP Worksheet #17
DRMOS-SB35-3	Soil	TBD[1]	TPH-e	1	SAP Worksheet #17
DRMOS-SB35-4	Soil	8 - 13	TPH-e	1	SAP Worksheet #17
DRMOS-SB36-1	Soil	0 – 0.5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB36-2	Soil	1 – 5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB36-3	Soil	TBD[1]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB36-4	Soil	8 - 11	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB36-5	Water	TBD[2]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB37-1	Soil	0 – 1	TPH-e, PCBs, Metals	1	SAP Worksheet #17

SAP Worksheet #18 – continued

DRMO South

Sampling Location/ID Number	Matrix	Depth[1] (Feet)	Analytical Group	Number of Samples	Sampling SOP Reference
DRMOS-SB37-2	Soil	1 – 5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB37-3	Soil	TBD[1]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB37-4	Soil	8 - 11	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB37-5	Water	TBD[2]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB38-1	Soil	0 – 0.5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB38-2	Soil	1 – 5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB38-3	Soil	TBD[1]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB38-4	Soil	8 - 13	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB39-1	Soil	0 – 0.5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB39-2	Soil	1 – 5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB39-3	Soil	TBD[1]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB39-4	Soil	8 - 13	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB39-5	Water	TBD[2]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB40-1	Soil	0 – 0.5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB40-2	Soil	1 – 5	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB40-3	Soil	TBD[1]	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB40-4	Soil	8 - 11	TPH-e, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB41-1	Soil	0 – 0.5	TPH-e	1	SAP Worksheet #17
DRMOS-SB41-2	Soil	1 – 5[3]	TPH-e	1	SAP Worksheet #17
DRMOS-SB41-3	Soil	1 - 5[4]	TPH-e	1	SAP Worksheet #17
DRMOS-SB42-1	Soil	0 – 0.5	TPH-e, PAHs, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB42-2	Soil	1 – 5	TPH-e, PAHs, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB42-3	Soil	TBD[1]	TPH-e, PAHs, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB42-4	Water	TBD[2]	TPH-e, PAHs, PCBs, Metals	1	SAP Worksheet #17
DRMOS-SB43-1	Soil	0 – 0.5	Metals	1	SAP Worksheet #17
DRMOS-SB43-2	Soil	1 – 5	Metals	1	SAP Worksheet #17

SAP Worksheet #18 – continued

Notes:

All results for soil will be reported on a dry-weight basis. Exact depth of the deeper samples will be determined in the field based on photoionization detector readings and visual clues.

1. To be collected from the capillary fringe, immediately above the water table. The water table is assumed to be encountered between 6 and 8 feet bgs but will be verified in the field and noted in the boring log and field logbook.
2. To be determined based on observed depth of the water table and the final screening interval.
3. To be collected from the top of the given depth interval.
4. To be collected from the bottom of the given depth interval.

DRMOS	Defense Reutilization and Marketing Office South
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
TPH-e	total petroleum hydrocarbons-extractable

SAP Worksheet #18 – continued

CTA North

Sampling Location/ID Number	Matrix	Depth[1] (Feet)	Analytical Group	Number of Samples	Sampling SOP Reference
CTAN-SB14-1	Soil	0 – 0.5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB14-2	Soil	1 – 5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB14-3	Soil	TBD[1]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB14-4	Soil	8 – 10[2]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB14-5	Water	TBD[3]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB15-1	Soil	0 – 0.5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB15-2	Soil	1 – 5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB15-3	Soil	TBD[1]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB15-4	Soil	8 – 10[2]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB16-1	Soil	0 – 0.5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB16-2	Soil	1 – 5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB16-3	Soil	TBD[1]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB16-4	Soil	8 – 10[2]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB16-5	Water	TBD[3]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB17-1	Soil	0 – 0.5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB17-2	Soil	1 – 5	PAHs, Metals, TPH-e	1	SAP Worksheet #17
CTAN-SB17-3	Soil	TBD[1]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB17-4	Soil	8 – 10[2]	PAHs, Metals, TPH-e, PCBs	1	SAP Worksheet #17
CTAN-SB18-1	Soil	0 – 0.5	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB18-2	Soil	1 – 5	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB18-3	Soil	TBD[1]	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB18-4	Soil	8 – 10[2]	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB19-1	Soil	0 – 0.5	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB19-2	Soil	1 – 5	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB19-3	Soil	TBD[1]	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB19-4	Soil	8 – 10[2]	TPH-e, Metals	1	SAP Worksheet #17

SAP Worksheet #18 – continued

CTA North

Sampling Location/ID Number	Matrix	Depth[1] (Feet)	Analytical Group	Number of Samples	Sampling SOP Reference
CTAN-SB20-1	Soil	0 – 0.5	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB20-2	Soil	1 – 5	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB20-3	Soil	TBD[1]	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB20-4	Soil	8 – 10[2]	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB20-5	Water	TBD[3]	TPH-e, Metals	1	SAP Worksheet #17
CTAN-SB21-1	Soil	0 – 0.5	TPH-e, PAHs, Metals	1	SAP Worksheet #17
CTAN-SB21-2	Soil	1 – 5	TPH-e, PAHs, Metals	1	SAP Worksheet #17
CTAN-SB22-1	Soil	0 – 0.5	TPH-e, PAHs	1	SAP Worksheet #17
CTAN-SB22-2	Soil	1 – 5	TPH-e, PAHs	1	SAP Worksheet #17
CTAN-SB23-1	Soil	0 – 0.5	TPH-e, PAHs, Metals	1	SAP Worksheet #17
CTAN-SB23-2	Soil	1 – 5	TPH-e, PAHs, Metals	1	SAP Worksheet #17
CTAN-SB24-1	Soil	0 – 0.5	PAHs	1	SAP Worksheet #17
CTAN-SB24-2	Soil	1 – 5	PAHs	1	SAP Worksheet #17
CTAN-SB25-1	Soil	2 – 2.5	TPH-e	1	SAP Worksheet #17
CTAN-SB25-2	Soil	5 – 5.5	TPH-e	1	SAP Worksheet #17
CTAN-SB25-3	Soil	8 – 8.5	TPH-e	1	SAP Worksheet #17

Notes:

All results for soil will be reported on a dry-weight basis. Exact depth of the deeper samples will be determined in the field based on photoionization detector readings and visual clues.

1. To be collected from the capillary fringe, immediately above the water table. The water table is assumed to be encountered between 6 and 8 feet bgs but will be verified in the field and noted in the boring log and field logbook.
2. To be collected below the 1925 to 1942 fill material layer.
3. To be determined based on observed depth of the water table and the final screening interval.

CTAN	Crane Test Area North
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl

SAP Worksheet #18 – continued

SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
TPH-e	Total petroleum Hydrocarbon-extractable

SAP Worksheet #19 — Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method	Containers	Sample volume	Preservation Requirements	Maximum Holding Time (preparation / analysis)
Water	PAHs	3520C/8270-SIM	2 x 1-Liter Amber Glass	1-Liter	2 to 6°C	7 days / 40 days
Water	Metals[1]	3010A/6020A	1 x 250 ml HDPE	50 ml	HNO_3, 2 to 6°C	180 days
Water	Mercury[1]	7470A	1 x 250 ml HDPE	50 ml	HNO_3, 2 to 6°C	28 days
Water	TPH-e	3520C/8015B	2 x 1-Liter Amber Glass	1-Liter	2 to 6°C	7 days / 40 days
Water	PCBs	3520C/8082	2 x 1-Liter Amber Glass	1-Liter	2 to 6°C	7 days / 40 days
Soil	TPH-e	3550B/8015B	2” diameter by 8” long, Acetate Sleeve	30-grams	2 to 6°C	14 days / 40 days
Soil	PAHs	3550B/8270C-SIM		30-grams	2 to 6°C	14 days / 40 days
Soil	PCBs	3550B/8082		30-grams	2 to 6°C	14 days / 40 days
Soil	Metals	3050B/6010B		5-grams	2 to 6°C	180 days
Soil	Mercury	7471A		6-grams	2 to 6°C	28 days

Notes:

1 Groundwater for metals analysis will be filtered in the laboratory with a 0.45 micron filter.

°C degrees Centigrade
HDPE high density polyethylene
HNO_3 nitric acid
ml milliliter
PAH polycyclic aromatic hydrocarbon
PCB polychlorinated biphenyl
SOP Standard Operating Procedure
SIM selected ion monitoring
TPH-e total petroleum hydrocarbons-extractable

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SAP Worksheet #20 — Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/ MSDs	No. of Source Water Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of PT Samples	Total No. of Samples to Lab
Soil	Metals (including mercury)	59	0	5% or 1 per 20 samples	1 per lot of water used to collect equipment rinsate samples (estimated 1 total)	1 per day for all reusable sampling equipment (estimated 6 total)	0	0	69
Soil	PAHs	31	0				0	0	40
Soil	TPH-e	79	0				0	0	90
Soil	PCBs	31	0				0	0	40
Groundwater	Metals (including mercury)	7	10% or 1 per 10 samples	1[a]		1 per day for all reusable sampling equipment (estimated 1 total)	0	0	10
Groundwater	PAHs	3		1[a]			0	0	6
Groundwater	TPH-e	7		1[a]			0	0	10
Groundwater	PCBs	6		1[a]			0	0	9

Notes:

[a] One of the primary samples will be submitted to the analytical laboratory with three times the standard volume of water. This sample will be identified to have the matrix spike (MS) and matrix spike duplicate (MSD) analyses performed; however, these samples are not considered separate.

MS/MSD matrix spike/matrix spike duplicate
PAH polycyclic aromatic hydrocarbon
PCB polychlorinated biphenyl
PT proficiency testing
TPH-e total petroleum hydrocarbon-extractable
VOA volatile organic analysis

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SAP Worksheet #21 — Project Sampling SOP References Table

No stand-alone SOPs have been referenced in this SAP. All project-specific tasks and procedures are detailed within SAP Worksheet #14.

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SAP Worksheet #22 — Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference	Comments
PID	Calibration with 100 parts per million Isobutylene	Daily	Within manufacturer's recommended value	According to manufacturer's instructions	Sampling personnel or health and safety representative	Manufacturer's instructions	
Water quality meter	Calibration with standards for pH, turbidity, specific conductance, oxidation/reduction potential, and dissolved oxygen per manufacturers' recommendation	Daily	Within manufacturer's recommended value	According to manufacturer's instructions	Sampling personnel	Manufacturer's instructions	

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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)
Vol. SVOC, Sec. 5.1	Total Extractable Hydrocarbons 'TEH' by GC/FID Method, EPA 8015B and 8015D; Rev 17, 11/30/11	Definitive	Water/Soil TPH-e	GC/FID	Curtis & Tompkins	N
Vol. SVOC, Sec. 8.2	Polynuclear Aromatic Hydrocarbons and 1,4-Dioxane by EPA 8270-SIM; Rev 5, 2/22/2013	Definitive	Water/Soil PAH-SIM	GC/MS	Curtis & Tompkins	N
Vol. Metals, Sec. 4.4	ICP Metals Analysis, Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES), Methods EPA 6010 & EPA 2007; Rev 14, 3/9/2012	Definitive	Soil Metals	ICP-AES	Curtis & Tompkins	N
Vol. Trace Metals, Sec. 4.6	Metals Analysis by ICP-MS, EPA 6020 & EPA 200.8; Rev 9, 3/9/2012	Definitive	Water Metals	ICP-MS	Curtis & Tompkins	N
Vol. Metals, Sec. 5.2	Digestion and Analysis of Solid Samples for Mercury Analysis, EPA 7471; Rev 17, 3/9/2012	Definitive	Soil Mercury	Cold Vapor	Curtis & Tompkins	N
Vol. Metals, Sec. 5.1	Digestion and Analysis of Aqueous Samples for Mercury, EPA 7470/EPA 245.1; Rev 16, 3/9/2012	Definitive	Water Mercury	Cold Vapor	Curtis & Tompkins	N
Vol. SVOC, Sec. 3.2	Polychlorinated Biphenyls, EPA 8082 & EPA 608; Rev 10, 4/19/2013	Definitive	Water/Soil PCBs	GC	Curtis & Tompkins	N
Vol. SVOC, Sec. 8.1	Semivolatile Organic Compounds (Base/Neutrals & Acids by GC/MS), EPA 8270C, 8270D, & EPA 625; Rev 12, 11/30/2011	Not Applicable (Maintenance)	Maintenance of GC/MS	GC/MS	Curtis & Tompkins	N

Notes:

1 Laboratory SOPs referenced are included as Attachment 3.

2 Note that there are no variances to the Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2.

AES atomic emission spectroscopy
ECD electron capture detector
FID flame ionization detector
GC gas chromatograph
GC/MS gas chromatograph / mass spectrometer
ICP inductively coupled plasma
PAH polynuclear aromatic hydrocarbon
PCB polychlorinated biphenyl
SIM selective ion monitoring
TPH total petroleum hydrocarbons

SAP Worksheet #24 — Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Check of mass spectral ion intensities (tuning procedure) using DFTPP	Prior to ICAL and at the beginning of each 12-hour period	Refer to method/SOP for specific ion criteria (meets SW-846 8270 m/z ratio requirements)	Retune instrument and verify	Analyst	Vol. SVOC, Sec. 8.2
GC/MS	Breakdown check (DDT) and peak tailing check	Prior to initial calibration and calibration verification, every 12 hours of analysis time	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown/peak tailing check.	Analyst	Vol. SVOC, Sec. 8.2 / DoD QSM (Version 4.2)
GC/MS	Initial Calibration (ICAL)	Initial calibration prior to sample analysis (5-point minimum)	Average RF for SPCCs ≥ 0.050 and RSD for RFs for CCCs < 30% and one option below: 1) linear – mean RSD for all analytes ≤15% 2) linear – least squares regression r ≥ 0.995, when RSD >15% 3) non-linear – COD (r^2) > 0.990 (6 points shall be used for second order, 7 points shall be used for third	Correct problem then repeat ICAL	Analyst	Vol. SVOC, Sec. 8.2 / DoD QSM (Version 4.2)

SAP Worksheet 24 continued

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Second source calibration verification (ICV)	Once after each initial calibration	Each analyte within ± 20% of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst	Vol. SVOC, Sec. 8.2 / DOD QSM (Version 4.2)
GC/MS	Continuing calibration verification (CCV)	Daily, before sample analysis, and every 12 hours of analysis time	RRF ≥ 0.050; and target analytes ≤ 20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration).	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst	Vol. SVOC, Sec. 8.2 / DOD QSM (Version 4.2)
GC/MS	Internal standards verification	Each field sample, standard, and QC sample	Retention time ± 30 seconds from retention time of the mid-point std. in the ICAL. EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst	Vol. SVOC, Sec. 8.2 / DOD QSM (Version 4.2)
GC/MS	Evaluation of relative retention time	Each sample	Relative retention time (RRT) of each target analyte within ± 0.06 RRT units of last RRT	Correct problem, then rerun ICAL	Analyst	Vol. SVOC, Sec. 8.2 / DOD QSM (Version 4.2)

SAP Worksheet 24 continued

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC	ICAL	Initial calibration prior to sample analysis and as needed	One of the following options: 1) RSD for all analytes ≤20% 2) linear – least squares regression r ≥ 0.995 3) non-linear – COD (r^2) > 0.990 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem, then rerun ICAL	Analyst	Vol. SVOC, Sec. 5.1 / Vol. SVOC, Sec. 3.2 / DoD QSM (Version 4.2)
GC	ICV	Once immediately after each initial calibration	All project analytes within established retention time windows GC Methods: All project analytes within ± 20% of expected value from ICAL HPLC / IC Methods: All project analytes within ± 15% of expected value from ICAL	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst	Vol. SVOC, Sec. 5.1 / Vol. SVOC, Sec. 3.2 / DoD QSM (Version 4.2)

SAP Worksheet 24 continued

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC	CCV	Daily, before sample analysis, after every 10 field samples, and at the end of analysis sequence.	All project analytes within established retention time windows GC Methods: All project analytes within ± 20% of expected value from ICAL HPLC / IC Methods: All project analytes within ± 15% of expected value from ICAL	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst	Vol. SVOC, Sec. 5.1 / Vol. SVOC, Sec. 3.2 / DoD QSM (Version 4.2)
ICP-AES	ICAL (minimum of three standards and a calibration blank)	Daily prior to sample analysis	If more than one calibration standard is used, r ≥ 0.995	Correct problem then repeat ICAL.	Analyst	Vol. Metals, Sec. 4.4 / DoD QSM (Version 4.2)
ICP-AES	Low level calibration check standard	Once after each ICAL	Value of all project analytes within ± 20% of true value	Correct problem then re-analyze.	Analyst	Vol. Metals, Sec. 4.4 / DoD QSM (Version 4.2)
ICP-AES	ICV	Once after each ICAL, prior to beginning a sample run	Value of all project analytes within ± 10% of true value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst	Vol. Metals, Sec. 4.4 / DoD QSM (Version 4.2)

SAP Worksheet 24 continued

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP-AES	CCV	After every 10 field samples, and at the end of analysis sequence.	Value of all project analytes within ± 10% of true value	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst	Vol. Metals, Sec. 4.4 / DoD QSM (Version 4.2)
ICP-MS	ICAL (minimum of one high standard and a calibration blank)	Daily prior to sample analysis	If more than one calibration standard is used, r ≥ 0.995	Correct problem then repeat ICAL.	Analyst	Vol. Trace Metals, Sec. 4.6 / DoD QSM (Version 4.2)
ICP-MS	Low level calibration check standard	Once after each ICAL	Value of all project analytes within ± 20% of true value	Correct problem then re-analyze.	Analyst	Vol. Trace Metals, Sec. 4.6 / DoD QSM (Version 4.2)
ICP-MS	ICV	Once after each ICAL, prior to beginning a sample run	Value of all project analytes within ± 10% of true value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst	Vol. Trace Metals, Sec. 4.6 / DoD QSM (Version 4.2)
ICP-MS	CCV	After every 10 field samples, and at the end of analysis sequence.	Value of all project analytes within ± 10% of true value	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst	Vol. Trace Metals, Sec. 4.6 / DoD QSM (Version 4.2)

SAP Worksheet 24 continued

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
CVAA	ICAL (minimum of five standards and a calibration blank)	Daily prior to sample analysis	If more than one calibration standard is used, r ≥ 0.995	Correct problem then repeat ICAL.	Analyst	Vol. Metals, Sec. 5.2 / Vol. Metals, Sec. 5.1 / DoD QSM (Version 4.2)
CVAA	ICV	Once after each ICAL	Value of all project analytes within ± 10% of true value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst	Vol. Metals, Sec. 5.2 / Vol. Metals, Sec. 5.1 / DoD QSM (Version 4.2)

SAP Worksheet 24 continued

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
CVAA	CCV	After every 10 field samples, and at the end of analysis sequence.	Value of all project analytes within ± 20% of true value	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst	Vol. Metals, Sec. 5.2 / Vol. Metals, Sec. 5.1 / DoD QSM (Version 4.2)

Notes:

AES	atomic emission spectroscopy
CA	corrective action
CCC	calibration check compound
CCV	continuing calibration verification
COD	coefficient of determination
CVAA	cold vapor atomic absorption
DDT	2,2-bis(p-chlorophenyl)-1,1,1-trichloroethane/dichlorodiphenyl-trichloroethane/p,p'-DDT
DFTPP	decafluorotriphenylphosphine
DoD QSM	Department of Defense Quality Systems Manual
EICP	extracted ion current profile
GC/MS	Gas chromatograph mass spectrometer
HPLC	high performance liquid chromatography
ICAL	initial calibration
ICP	inductively coupled plasma
ICV	second source calibration verification
RSD	relative standard deviation
RF	response factor
SOP	Standard Operating Procedure
SPCC	system performance check compound
SVOC	semivolatile organic compound

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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
GC	Replace disposables and maintain instrument and detectors per SOP and instrument manual (e.g., replace septa and liners, leak check and check tightness of the capillary column detector, clean autosampler syringes, replace vials)	Preventative maintenance	The instrument operator performs daily checks per lab SOP and quality manual	Daily when in use and per lab maintenance schedule	CCV passes criteria	Investigate and correct problem and recalibrate instrument	Analyst / Department Manager	Vol. SVOC, Sec. 5.1 / Vol. SVOC, Sec. 3.2
GC/MS	Replace disposables and maintain instrument and detectors per SOP and instrument manual (e.g., replace septa and liners, replace guard column, replace blue seal, trim analytical column, clean the source)	Preventative maintenance	The instrument operator performs daily checks per lab SOP and quality manual	Daily when in use and per lab maintenance schedule	CCV passes criteria	Investigate and correct problem and recalibrate instrument	Analyst / Department Manager	Vol. SVOC, Sec. 8.1
ICP-AES	Replace disposables and maintain instrument per SOP and instrument manual (e.g., replenish rinse-water reservoir, change the pump windings, change the sample tubing,clean/change	Preventative maintenance	The instrument operator performs daily checks per lab SOP and quality manual	Daily when in use and per lab maintenance schedule	CCV passes criteria	Investigate and correct problem and recalibrate instrument	Analyst / Department Manager	Vol. Metals, Sec. 4.4

SAP Worksheet #25 – continued

Instrument Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
	torch and injector, lubricate autosampler tracks)							
ICP-MS	Replace disposables and maintain instrument per SOP and instrument manual (e.g., replenish rinse-water reservoir, empty/rinse drain vessel, change the sample and internal standard pump windings, change drain pump winding)	Preventative maintenance	The instrument operator performs daily checks per lab SOP and quality manual	Daily when in use and per lab maintenance schedule	CCV passes criteria	Investigate and correct problem and recalibrate instrument	Analyst / Department Manager	Vol. Trace Metals, Sec. 4.6 / DoD QSM (Version 4.2)
CVAA	Replace disposables and maintain instrument per SOP and instrument manual (e.g., replace pump windings, clean drying tube/gas-liquid separator, replace lamp)	Preventative maintenance	The instrument operator performs daily checks per lab SOP and quality manual	Daily when in use and per lab maintenance schedule	CCV passes criteria	Investigate and correct problem and recalibrate instrument	Analyst / Department Manager	Vol. Metals, Sec. 5.2 / Vol. Metals, Sec. 5.1

Notes:
AES – atomic emission spectroscopy
CCV – continuing calibration verification
CVAA – cold vapor atomic absorption
GC – gas chromatograph
ICP – inductively coupled plasma
MS – mass spectrometer
SOP – standard operating procedure

SAP Worksheet #26 — Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): ARCADIS field team, supervised by the ARCADIS Field Team Lead
Sample Packaging (Personnel/Organization): ARCADIS field team, supervised by the ARCADIS Field Team Lead
Coordination of Shipment (Personnel/Organization): ARCADIS Project Chemist or ARCADIS Project Engineer
Type of Shipment/Carrier: Laboratory (Curtis & Tompkins) courier or commercial carrier
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Laboratory (Curtis & Tompkins) sample custodian
Sample Custody and Storage (Personnel/Organization): Laboratory (Curtis & Tompkins) sample custodian
Sample Preparation (Personnel/Organization): Laboratory (Curtis & Tompkins) sample preparation group
Sample Determinative Analysis (Personnel/Organization): Laboratory (Curtis & Tompkins) bench chemist
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 3 months
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extraction and digestion must be conducted according to the requirements specified in SAP Worksheet #19
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL
Personnel/Organization: Laboratory (Curtis & Tompkins) sample custodian
Number of Days from Analysis: Approximately 60 days

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SAP Worksheet #27 — Sample Custody Requirements Table

27.1 Sample Number

All samples (including field QC samples) submitted to the off-site analytical laboratory will be uniquely numbered. The soil and groundwater samples will be identified by the following formats, following the sample identification scheme used for the site inspection at DRMO South and CTA North (Trevet 2012):

Soil and Groundwater within the DRMO South and CTA North

DRMOS-SBXX-X

Example: "DRMOS-SB01-1"; where "DRMOS" is the site name, "SB01" is the soil boring number 1, and "1" is the first sample collected at that boring. Site names for this investigation are DRMOS (DRMO South) and CTAN (CTA North). The soil boring numbers will start at SB27 for DRMO South and SB14 for CTA North.

Field Quality Control Samples

Field QC samples will be identified as "DRMOS-XXX," where "DRMOS" is the site name and "XXX" is a sequential number starting with "001."

Sample identification numbers are listed on SAP Worksheet #18. The sample number will be recorded in the field logbook, on the sample container labels at the time of sample collection, and on the COC form.

27.2 Sample Labeling

Correct sample labeling and the corresponding notation of the sample identification numbers in the field logbook and on the COC forms will be utilized to prevent misidentification of samples and the sample results. All sample labels will be completed legibly with indelible ink. The labels will be affixed to the sample container at the time of sample collection. The sample labels will include the following, at a minimum:

- Sample identification number;
- Date and time of collection;
- Project name or number;
- Company name;
- Name/initials of the collector;
- Sample location and depth;
- Analysis required; and
- Preservatives added, if any.

A copy of a sample label is included in Attachment 1.

SAP Worksheet #27 – continued

27.3 Sample Handling and Shipping

Sample containers (as specified in SAP Worksheet #19) will be placed in a resealable plastic bag to protect the sample from moisture and to prevent breakage and potential cross-contamination during transportation to the laboratory. All glass sample containers will be protected with bubble wrap first, then placed in resealable bags if transported by a commercial carrier. Each cooler will be shipped with a temperature blank. A temperature blank is a container filled with tap water and stored in the cooler during sample collection and transportation. The temperature of the cooler will be recorded by the laboratory on the COC record immediately upon receipt of the samples. Custody of samples must be maintained throughout the shipment of samples to the selected laboratory. The following procedures will be used to send samples to be analyzed by the laboratory:

- Pertinent information on the sample labels will be filled out, the sample will be placed in a new sample container, and the container lid will be closed tightly.
- Filled sample containers will be enclosed in clear plastic bags through which sample labels are visible, and the bags will be sealed.
- Sample containers will be placed upright in a cooler in such a way that they do not and will not touch during shipment.
- Samples to be shipped by commercial carrier will be packed in a sample cooler lined with a plastic bag.
- Only waterproof, high-strength plastic ice chests or coolers will be used to ship samples.
- Cooler drains will be taped closed prior to sample shipment.
- Bags of ice will be placed around, among, and on top of the sample containers.
- Copies of the COC record(s) will be sealed in a resealable bag and then taped to the inside of the cooler lid.
- Cooler lids will be secured by taping prior to shipment. The cooler will be wrapped completely with strapping tape at a minimum of two locations.
- Two custody seals will be taped across the cooler lid: one seal in the front and one seal in the back. Clear tape will be applied to the custody seals to prevent accidental breakage during shipment. A copy of a sample COC seal is included in Attachment 1.
- The pouch for the airbill will be placed on the cooler and secured with clear tape. A completed shipping label will be taped to the top of the cooler. The shipping label will have a return address.
- The cooler will be shipped by overnight express or courier to the laboratory. Samples will be shipped with sufficient ice to keep samples cool for up to three days to allow for shipment delays.

SAP Worksheet #27 – continued

- Saturday deliveries will be coordinated with the laboratory in advance, and field sampling personnel or their designee will ensure that Saturday delivery stickers are placed on each cooler by the commercial courier.

27.4 Field Documentation

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 COC documentation. All documentation will be recorded in a field logbook in indelible ink.

27.4.1 Chain of Custody

ARCADIS will use standard sample custody procedures to maintain and document sample integrity during collection, transportation, storage, and analysis. A sample will be considered to be in custody if one of the following statements applies:

- It is in a person's physical possession or view.
- It is in a secure area with restricted access.
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

COC procedures are used to document and track samples from collection through reporting of analytical results and to serve as permanent records of sample handling and shipment. Strict COC protocol will be maintained for all samples collected during this project. The COC forms will be filled out with indelible ink by the ARCADIS Field Team Lead, and any mistakes made will be crossed out with a single line and initialed and dated. COC forms will be completed for every cooler and will be sealed in a resealable bag and taped to the inside of the lid of the cooler. COC forms will include the following information:

- Project name and number,
- Project contact information (both ARCADIS and laboratory),
- Sampling location,
- Name and signature of sampler,
- Destination of samples (laboratory name),
- Unique sample identification number,
- Matrix type,
- Matrix spike/matrix spike duplicate samples,
- Date and time of collection,

SAP Worksheet #27 – continued

- Number and type of containers filled,
- Analysis requested,
- Preservatives used (if applicable),
- Filtering (if applicable),
- Sample designation (grab or composite),
- Sampler's signature,
- "Relinquished By"- information (signature, company, date/time),
- "Received By"- information (signature, company, date/time),
- Data package requirement (Level III or IV),
- Airbill number (if applicable), and
- Remarks.

Unused lines on the COC record will be crossed out. COC records that are initiated in the field will be signed by field personnel. The COC will be placed in a waterproof plastic bag and taped to the inside of the shipping container. Signed airbills will serve as evidence of custody transfer between field personnel and the courier, and between the courier and the laboratory. Copies of the COC record and the airbill will be retained and filed by field personnel before the containers are shipped. A copy of a sample COC form is included in Attachment 1.

The laboratory will provide a secure storage area for all samples. Access to this area will be restricted to authorized personnel. The custodian will ensure that samples requiring special handling, including samples that are heat- or light-sensitive, radioactive, or have other unusual physical characteristics, will be properly stored and maintained prior to analysis.

27.4.2 Field Logbooks

A permanently bound field logbook with consecutively numbered pages, used for sampling activities only, will be assigned to this project. All entries will be recorded in indelible ink. The logbook pages will be signed by the responsible sampler at the end of each workday, and any unused portions of the logbook pages will be crossed out, signed, and dated.

If it is necessary to transfer the logbook to another person, the person relinquishing the logbook will sign and date the last page used, and the person receiving the logbook will sign and date the next page to be used.

At a minimum, the logbook will contain the following information:

- Descriptions of deviations from this SAP
- Problems encountered and corrective action taken

SAP Worksheet #27 – continued

- Identification of field QC samples
- Project name and location
- Date and time
- Personnel in attendance
- General weather information
- Work performed
- Field observations
- Sampling performed, including specifics such as location, type of sample, types of analyses, and sample identification
- Field analyses performed, including results, instrument checks, problems, and calibration records for field instruments
- QC activities
- Verbal or written instructions
- Any other events that may affect the samples

27.4.3 Document Corrections

Changes or corrections on any project documentation will be made by crossing out the erroneous item with a single line, initialing (by the person performing the correction), and dating the correction. The original item, although erroneous, must remain legible beneath the cross-out line. The new information should be written clearly above the crossed-out item.

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SAP Worksheet #28 — Laboratory QC Samples Table

Matrix: Soil/Water
Analytical Group: PAHs
Analytical Method / SOP Reference: USEPA 8270C-SIM / Vol. SVOC, Sec. 8.2

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 analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > QL. Blank result must not otherwise affect sample results.	Determine cause of contamination and re-prep and reanalyze method blank and all samples processed with the non-conforming method blank.	Analyst	Accuracy/Bias - Contamination	No analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > QL. Blank result must not otherwise affect sample results.
Surrogate	Every analytical sample	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits	Correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst	Accuracy/Bias	Acceptance criteria by DoD QSM 4.2, if available or otherwise lab in-house control limits (Attachment 4)
LCS or LCS/LCSD pair if insufficient sample for MS/MSD	One per sample preparation batch	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits	Correct problem then re-prep and reanalyze LCS and all samples processed with the non-conforming LCS.	Analyst	Accuracy/Bias	Acceptance criteria by DoD QSM 4.2, if available, or otherwise lab in-house control limits (Attachment 4)

SAP Worksheet #28 – continued

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 every 20 project samples per matrix	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available, otherwise use in-house LCS control limits	If result is indicative of matrix interference, discuss in case narrative. Otherwise check for possible source of error, and re-extract / reanalyze the sample.	Analyst	Interferences - Accuracy/Bias - Precision	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available. Otherwise, use in-house LCS control limits (Attachment 4)

SAP Worksheet #28 – continued

Matrix: Soil/Water
Analytical Group: TPH-e
Analytical Method / SOP Reference: USEPA 8015B / Vol. SVOC, Sec. 5.1

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 analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > QL. Blank result must not otherwise affect sample results.	Determine cause of contamination and re-prep and reanalyze method blank and all samples processed with the non-conforming method blank.	Analyst	Accuracy/Bias - Contamination	No analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > QL. Blank result must not otherwise affect sample results.
Surrogate	Every analytical sample	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits	Correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst	Accuracy/Bias	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits (Attachment 4)
LCS or LCS/LCSD pair if insufficient sample for MS/MSD	One per sample preparation batch	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits	Correct problem then re-prep and reanalyze LCS and all samples processed with the non-conforming LCS.	Analyst	Accuracy/Bias	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits (Attachment 4)

SAP Worksheet #28 – continued

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 every 20 project samples per matrix	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available, otherwise use in-house LCS control limits.	If result is indicative of matrix interference, discuss in case narrative. Otherwise check for possible source of error, and re-extract / reanalyze the sample.	Analyst	Interferences - Accuracy/Bias - Precision	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available, otherwise use in-house LCS control limits (Attachment 4).

SAP Worksheet #28 – continued

Matrix: Soil/Water
Analytical Group: PCBs
Analytical Method / SOP Reference: USEPA 8082 / Vol. SVOC, Sec. 3.2

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 analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > QL. Blank result must not otherwise affect sample results.	Determine cause of contamination and re-prep and reanalyze method blank and all samples processed with the non-conforming method blank.	Analyst	Accuracy/Bias - Contamination	No analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > QL. Blank result must not otherwise affect sample results.
Surrogate	Every analytical sample	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits	Correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst	Accuracy/Bias	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits (Attachment 4)
LCS or LCS/LCSD pair if insufficient sample for MS/MSD	One per sample preparation batch	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits	Correct problem then re-prep and reanalyze LCS and all samples processed with the non-conforming LCS.	Analyst	Accuracy/Bias	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits (Attachment 4)

SAP Worksheet #28 – continued

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 every 20 project samples per matrix	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available, otherwise use in-house LCS control limits.	If result is indicative of matrix interference, discuss in case narrative. Otherwise check for possible source of error, and re-extract / reanalyze the sample.	Analyst	Interferences - Accuracy/Bias - Precision	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available, otherwise use in-house LCS control limits (Attachment 4).

SAP Worksheet #28 – continued

Matrix: Soil/Water
Analytical Group: Metals, Mercury
Analytical Method / SOP Reference: USEPA 6020A / Vol. Trace Metals, Sec. 4.6, EPA 6010B / Vol. Metals, Sec. 4.4, EPA 7470 / Vol. Metals, Sec. 5.1, EPA 7471 / Vol. Metals, Sec. 5.2

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 analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > RL. Blank result must not otherwise affect sample results.	Correct problem then re-prep and reanalyze blank and all samples processed with the non-conforming blank.	Analyst	Accuracy/Bias - Contamination	No analytes detected > ½ QL. For common laboratory contaminants, no analytes detected > RL. Blank result must not otherwise affect sample results.
LCS or LCS/LCSD pair if insufficient sample for MS/MSD	One per preparation batch	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits	Correct problem then re-prep and reanalyze LCS and all samples processed with the non-conforming LCS.	Analyst	Accuracy/Bias	Acceptance criteria specified by DoD QSM 4.2, if available, or otherwise lab in-house control limits (Attachment 4)

SAP Worksheet #28 – continued

Matrix: Soil/Water
Analytical Group: Metals, Mercury
Analytical Method / SOP Reference: USEPA 6020A / Vol. Trace Metals, Sec. 4.6, EPA 6010B / Vol. Metals, Sec. 4.4, EPA 7470 / Vol. Metals, Sec. 5.1, EPA 7471 / Vol. Metals, Sec. 5.2

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 every 20 project samples per matrix	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available, otherwise use in-house LCS control limits. RPD ≤ 20% (between MS and MSD).	If result is indicative of matrix interference, discuss in case narrative. Otherwise check for possible source of error, and re-extract / reanalyze the sample.	Analyst	Interferences - Accuracy/Bias - Precision	For matrix evaluation, use LCS acceptance criteria specified by DoD QSM 4.2, if available, otherwise use in-house LCS control limits (Attachment 4). RPD ≤ 20% (between MS and MSD).
Interference check solutions (ICS-AB)	At the beginning and end of an analytical run and every 8 hours	ICS-AB: Within ± 20% of true value	Terminate analysis, locate and correct problem, reanalyze ICS and all affected samples	Analyst	Accuracy	ICS-AB: Within ± 20% of true value
Dilution Test	Per sample preparation batch.	1:5 dilution must agree within ± 10% of the original determination	Perform post-digestion spike addition	Analyst	Accuracy/Bias	1:5 dilution must agree within ± 10% of the original determination
Analytical Spike	When dilution test fails or analyte concentration in all samples < 50x LOD	Recovery within 75-125% of expected value	Run all samples by method of standard addition (MSA)	Analyst	Interferences - Accuracy/Bias - Precision	Recovery within 75-125% of expected value

SAP Worksheet #28 – continued

Notes:

DoD	Department of Defense
DQI	data quality indicator
ICS	interference check solution
LCS/LCSD	laboratory control sample/ laboratory control sample duplicate
LOD	limit of detection
MS/MSD	matrix spike/matrix spike duplicate
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
QA	quality assurance
QL	quantitation limit
QSM	Quality Systems Manual
SAP	Sampling and Analyses Plan
SIM	selective ion monitoring
SOP	Standard Operating Procedure
SVOC	semivolatile organic compound
USEPA	United States Environmental Protection Agency

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SAP Worksheet #29 — Project Documents and Records Table

Document	Where Maintained
Field logbook	ARCADIS Project File
Field forms	ARCADIS Project File
Sample labels	Laboratory (Curtis & Tompkins)
COC form	ARCADIS Project File and Laboratory (Curtis & Tompkins) Project File
Supplies certification	ARCADIS Project File
Shipping records	ARCADIS Project File
Field audit and nonconformance reports	ARCADIS Project File
Laboratory data package including: Sample receipt and login Laboratory internal COC Instrument calibration logs Sample preparation logs Sample analysis/run logs Nonconformance reports including corrective actions	Laboratory (Curtis & Tompkins) Project File, ARCADIS Project File, and NAVFAC SW Administrative Record
Data validation report	Validator (LDC) Project File, ARCADIS Project File, and NAVFAC SW Administrative Record

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SAP Worksheet #30 — Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization
Soil (DRMO South and CTA North)	PAHs-SIM Metals Mercury TPH-e PCBs	All	USEPA 8270C-SIM USEPA 6010B USEPA 7471A USEPA 8015B USEPA 8082	21 days	Curtis & Tompkins Laboratories 2323 5th Street Berkeley, CA 94710 Contact: Will Rice (510) 204-2221	TestAmerica West Sacramento 880 Riverside Parkway West Sacramento, CA 95605 Contact: To Be Determined
Groundwater	PAHs-SIM Metals Mercury TPH-e PCBs	All	USEPA 8270C-SIM USEPA 6020A USEPA 7470A USEPA 8015B USEPA 8082	21 days		

Notes:

CTA	Crane Test Area
DRMO	Defense Reutilization and Marketing office
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
SIM	selective ion monitoring
TPH-e	total petroleum hydrocarbons-extractable
USEPA	United States Environmental Protection Agency

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SAP Worksheet #31 — Planned Project Assessments Table

Assessment Type	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 (CA)	Person(s) Responsible for Monitoring Effectiveness of CA
Field Readiness Review	Before mobilization for the project and before major phases of work are initiated	Internal	ARCADIS	ARCADIS PM	ARCADIS PM	ARCADIS PM	ARCADIS QAM or designee
Field Sampling Surveillance	Once at the beginning of field sampling	Internal	ARCADIS	ARCADIS Project Chemist	ARCADIS PM	ARCADIS PM	ARCADIS QAM or Project Chemist
Data Review Surveillance	Once for project when duration less than 6 months	Internal	ARCADIS	ARCADIS Project Chemist	ARCADIS Project Chemist	ARCADIS Project Chemist	ARCADIS QAM or designee

Notes:
PM Project Manager
QAM Quality Assurance Manager

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SAP Worksheet #32 — Assessment Findings and Corrective Action Responses

Assessment Type	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
Field Sampling Surveillance	Surveillance Report	ARCADIS PM	7 days after the inspection is complete	Corrective Action Report	ARCADIS PM and QAM/designee	5 days after notification
Data Review Surveillance	Surveillance Report	ARCADIS PM	7 days after the inspection is complete	Corrective Action Report	ARCADIS PM and QAM/designee	14 days after notification

Notes:
PM Project Manager
QAM Quality Assurance Manager

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SAP Worksheet #33 — QA Management Reports Table

Type of Report	Frequency	Projected Delivery Date(s)	Persons Responsible for Report Preparation	Report Recipient(s)
Field Sampling Surveillance Report	One at start up of sampling	Within 7 days of first day of sampling	ARCADIS Project Chemist	ARCADIS PM, Project Chemist, QAM
Data Review Surveillance Report	One after all data generated and reviewed	Within 7 days of receipt of final validation report	ARCADIS Project Chemist	ARCADIS PM, Project Chemist, QAM

Notes:
PM Project Manager
QAM Quality Assurance Manager

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SAP Worksheet #34 — Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (name, organization)
Field logbook	Field logbooks will be reviewed weekly and verified that the information is complete. The inspection will be documented in daily QC reports.	Internal	ARCADIS Field Team Lead
COC forms	COC forms will be reviewed daily when they are completed and verified for completeness.	Internal	ARCADIS Field Team Lead
Sample receipt	For samples shipped via courier or commercial carrier, the ARCADIS Project Chemist or Project Engineer will verify receipt of samples by the laboratory the day following shipment.	Internal	ARCADIS Project Chemist or Project Engineer
Sample logins	Sample login information will be reviewed and verified for completeness in accordance with the COC forms.	Internal External	ARCADIS Project Chemist Laboratory (Curtis & Tompkins) PM
Laboratory data prior to release	Laboratory data will be reviewed and verified for completeness against analyses requested on the COC forms.	External	Laboratory (Curtis & Tompkins) PM
Laboratory data due at turnaround time listed on COC	Laboratory data will be verified that the analyses reported are consistent with the analytical suite requested on the COC forms.	External	ARCADIS Project Chemist
Laboratory data packages	All laboratory data packages will be verified by the laboratory performing the work for completeness. Data packages will then be reviewed by the ARCADIS Project Chemist for completeness. Subsequently, data packages will be evaluated externally by undergoing data validation.	Internal External External	Laboratory (Curtis & Tompkins) ARCADIS Project Chemist Third-party data validators (Laboratory Data Consultants)
Field and electronic data	One hundred percent of manual entries will be reviewed against the hardcopy information and 10 percent of electronic uploads will be checked against the hardcopy.	External	ARCADIS Project Chemist

Notes:
COC chain of custody
PM Project Manager

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SAP Worksheet #35 — Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field logbook	Field logbooks will be reviewed weekly for accuracy associated with each sampling event. The inspection will be documented in daily QC reports.	ARCADIS PM (Hoa Voscott) or designee
IIa	COC forms	COC forms will be reviewed daily to ensure that project information, sample analyses requested, number of field QC samples collected, and percent level III or IV validation chosen is accurate and in accordance with the requirements in this SAP.	ARCADIS Project Chemist (Dennis Dyke)
IIa	Sample receipt	The sample cooler will be checked for compliance with temperature and packaging requirements.	Laboratory (Curtis & Tompkins) Sample Custodian
IIa	Sample logins	Sample login will be reviewed for accuracy against the COC form.	ARCADIS Project Chemist (Dennis Dyke) Laboratory (Curtis & Tompkins) PM or designee
IIa	Laboratory data prior to release	Laboratory data will be reviewed to ensure that the data is accurate and meets the requirements in this SAP. Prior to release, data will be validated as follows:	Laboratory (Curtis & Tompkins) PM or designee
IIa	Laboratory data prior to release	100% of the data comply with the method- and project-specific requirements; any deviations or failure to meet criteria are documented for the project file.	Laboratory (Curtis & Tompkins) Analyst
IIa	Laboratory data prior to release	100% of manual entries are free of transcription errors and manual calculations are accurate; computer calculations are spot-checked to verify program validity; data reported are compliant with method- and project-specific QC requirements; raw data and supporting materials are complete; spectral assignments are confirmed; descriptions of deviations from method or project requirements are documented; significant figures and rounding have been appropriately used; reported values include dilution factors; and results are reasonable.	Laboratory (Curtis & Tompkins) Peer Analyst
IIa	Laboratory data prior to release	Data reported comply with method- and project-specific QC requirements; the reported information is complete; the information in the report narrative is complete and accurate; and results are reasonable.	Laboratory (Curtis & Tompkins) Supervisor
IIa	Laboratory data prior to release	Data reported comply with method- and project-specific QC; analytical methods are performed in compliance with approved SOPs. This review may be conducted after release of data.	Laboratory (Curtis & Tompkins) Quality Assurance Manager

SAP Worksheet #35 – continued

Step IIa/IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Laboratory data due at turnaround time listed on COC	Laboratory data will be reviewed to ensure that the data reported met the analyte list and limits listed in Worksheet #15.	ARCADIS Project Chemist (Dennis Dyke)
IIa	Laboratory data packages	All laboratory data packages will be validated by the laboratory performing the work for technical accuracy before it is submitted.	Laboratory (Curtis & Tompkins) PM or designee
IIa	Laboratory data packages	Data packages will then be reviewed for accuracy against the laboratory data that was faxed or e-mailed at the turnaround time listed on the COC.	ARCADIS Project Chemist (Dennis Dyke)
IIa	Laboratory data packages	Data packages will be evaluated externally by undergoing data validation.	Third-party data validator, Laboratory Data Consultants
IIb	Data validation reports	Data validation reports will be reviewed in conjunction with the project DQOs and data quality indicators.	ARCADIS Project Chemist (Dennis Dyke)

Notes:
DQO Data quality objective
PQCM Project Quality Control Manager
QC Quality control
SAP Sampling and analysis plan
SOP Standard operating procedure

SAP Worksheet #36 — Analytical Data Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa	Soil	PAHs, TPH-e, Metals (including Mercury), PCBs	CLP NFG[1], EWI #1 (10% Level IV validation, 90% Level III validation)[2], USEPA SW 846[3], and this UFP-SAP as applicable	Validator (Laboratory Data Consultants) PM or designee
IIb	Soil	PAHs, TPH-e, Metals (including Mercury), PCBs	CLP NFG[1], EWI #1 (10% Level IV validation, 90% Level III validation)[2], USEPA SW 846[3], and this UFP-SAP as applicable	Validator (Laboratory Data Consultants) PM or designee
IIa	Water	PAHs, Metals (including Mercury), TPH-e, PCBs	CLP NFG[1], EWI #1 (10% Level IV validation, 90% Level III validation)[2], USEPA SW 846[3], and this UFP-SAP as applicable	Validator (Laboratory Data Consultants) PM or designee
IIb	Water	PAHs, Metals (including Mercury), TPH-e, PCBs	CLP NFG[1], EWI #1 (10% Level IV validation, 90% Level III validation)[2], USEPA SW 846[3], and this UFP-SAP as applicable	Validator (Laboratory Data Consultants) PM or designee

1 USEPA, Contract Laboratory Program National Functional Guidelines for Organic Methods Data Review (2008), Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (2010).

2 Southwest Division Naval Facilities Engineering Command, Environmental Work Instruction #1, 3EN2.1, Chemical Data Validation (2001).

3 USEPA SW 846, Third Addition, Test Methods for Evaluation Solid Waste, update 1, July 1992: update IIA, August 1993; update II, September 1994; update IIB, January 1995; update III, December 1996; update IIIA, April 1998

EWI	Environmental Work Instruction
PAH	polycyclic aromatic hydrocarbons
PCB	polychlorinated biphenyl
TPH-e	total petroleum hydrocarbons-extractable
PM	Project Manager
UFP-SAP	Uniform Federal Policy Sampling and Analysis Plan
CLP-NFG	Contract Laboratory Program National Functional Guidelines
USEPA	Unites States Environmental Protection Agency

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SAP Worksheet #37 — Usability Assessment

37.1 Measurement Quality Objectives for Chemical Data

All analytical results will be evaluated in accordance with precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) parameters to document the quality of the data and to ensure that the data are of sufficient quality to meet the project objectives. The following subsections describe each of the PARCCS parameters and how they will be assessed for this project.

37.1.1 Precision

Precision is a measure of the reproducibility among a set of replicate results or the agreement among repeat observations made under the same conditions. Analytical precision is the measurement of the variability associated with duplicate or replicate analyses. Field duplicate, laboratory duplicate, matrix spike/matrix spike duplicate (MS/MSD) pairs, and laboratory control sample/laboratory control sample duplicate (LCS/LCSD) pairs (if analyzed) will be used to assess field and analytical precision. The precision measurement is determined using the relative percent difference (RPD) between the duplicate sample results. The RPD is calculated according to the following formula:

$$\text{RPD} = \frac{(V1 - V2) \times 100}{(V1 + V2)/2}$$

where: V1 = first duplicate concentration
V2 = second duplicate concentration

The RPD limits for laboratory duplicates, MS/MSD pairs, and LCS/LCSD pairs are presented in Worksheet #28 and the RPD limits for field duplicates are listed in Worksheet #12.

37.1.2 Accuracy

Accuracy is the degree of agreement between an analytical measurement and a reference accepted as a true value. Field accuracy will be assessed by collecting and analyzing equipment rinsate and source water blank quality control (QC) samples as appropriate. These QC samples will be used to evaluate the potential for target analytes to enter samples as a result of sampling processes.

A program of sample spiking will be conducted to evaluate laboratory/analytical accuracy. This program includes analysis of the MS/MSD samples, LCSs or blank spikes, surrogate standards, and method blanks. MS/MSD samples will be prepared and analyzed at a frequency of 5 percent for groundwater samples. LCS or blank spikes are also analyzed at a frequency of 5 percent. Surrogate standards, where applicable, are added to every sample analyzed for organic constituents. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy according to the following formula:

$$\text{Percent Recovery} = \frac{S - C}{T} \text{ x } 100$$

where:

S = Measured spike sample concentration
C = Sample concentration
T = True or actual concentration of the spike

Accuracy goals for field and laboratory QC samples are presented in Worksheets #12 and #28, respectively. Results that fall outside the accuracy goals will be further evaluated based on the results of other QC samples.

37.1.3 Representativeness

Unlike precision and accuracy, which can be expressed in quantitative terms, representativeness is a qualitative parameter. Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. For this project, representative data will be obtained through careful selection of sampling locations and analytical parameters. Representative data will also be obtained through proper collection and handling of samples to avoid interference and minimize contamination. Representativeness of data will also be ensured through established field and laboratory procedures and their consistent application. To aid in evaluating the representativeness of the sample results, field blanks (if appropriate) and laboratory blank samples will be evaluated for the presence of contaminants. Data determined to be non-representative, by comparison with existing data, will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

37.1.4 Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP and when none of the QC criteria that affect data usability is exceeded. When all data validation is completed, the percent completeness value will be calculated by dividing the number of usable sample results by the total number of sample results planned for this investigation.

Completeness will also be evaluated as part of the data quality assessment process. This evaluation will help determine whether any limitations are associated with the decisions to be made based on the data collected. A completeness goal of 90 percent has been set for this project. If completeness goals are not achieved, the sources of non-conformances will be evaluated to determine whether re-sampling and re-analysis is necessary.

37.1.5 Comparability

Comparability is a qualitative parameter that expresses the confidence with which one data set can be compared to another. This is a concern when current data are being integrated with historical or future data. Comparability of data is maximized through the use of standard

operating procedures in the field and the laboratory, standardized analytical methods, and consistent units of measure.

37.1.6 Sensitivity

The method detection limit (DL) is the minimum concentration of an analyte that can be reliably distinguished from background noise for a specific analytical method. The quantitation limit (QL) represents the lowest concentration of an analyte that can be accurately and reproducibly quantified in a sample matrix. The QLs are typically several times the method DL to allow for matrix effects.

Sensitivity describes the relationship between the laboratory QLs and the project action limits (PALs), which are selected based on the project quality objectives (PQOs). Analytes and their QLs and PALs are presented in SAP Worksheet #15. Wherever practical, analytical methods for this project have been selected such that the QL for each target analyte is below the associated PAL, indicating that the selected analytical method is capable of quantifying analytes of concern at or below the selected PALs. QLs that exceed the PALs will be discussed further in the uncertainties section of the risk assessment. This discussion will use multiple lines of evidence to determine if the chemical should be considered further.

37.2 Data Quality Assessment

After data have been reviewed, verified, and validated in accordance with the procedures described in SAP Worksheets #34 through #36, the data must be further evaluated to determine if the project quality objectives (PQOs) have been met.

To the extent possible, ARCADIS will follow the United States Environmental Protection Agency's (USEPA's) data quality assessment (DQA) process to verify that the type, quality, and quantity of data collected are appropriate for their intended use. DQA methods and procedures are outlined in two USEPA guidance documents: Data Quality Assessment Statistical Tools for Practitioners (USEPA 2006b) and Data Quality Assessment, A Reviewer's Guide (USEPA 2006c). The DQA process consists of five steps: (1) review the PQOs and sampling design; (2) conduct a preliminary data review; (3) select a statistical test; (4) verify the assumptions of the statistical test; and (5) draw conclusions from the data. Note that there are cases in which the five-step DQA process cannot be followed completely due to the qualitative nature of the PQOs. In these cases, ARCADIS will systematically assess data quality and data usability. The following outlines and describes components of this assessment:

- A review of sample design and sampling methods to verify that the methods were implemented as planned and are adequate to support project objectives;
- A review of field QC samples and the adequacy of field decontamination procedures;
- A review of QLs to determine if acceptance criteria have been met;
- A review of data comparability, if appropriate;
- A review of PQOs to determine whether they have been achieved by the data collected; and

- A review of project-specific data quality indicators for PARCCS parameters.

The ARCADIS Project Chemist will be responsible for performing the data assessment for this project and will prepare a DQA report that will summarize the findings of the data assessment and discuss the usability of the data to be included in the report.

In the event that the laboratory analytical data are limited in use based on usability review and data validation, any limitation will be taken into consideration when comparing results to the PALs. Data qualifier flags are used by the laboratory and during data validation and assessment to notify the user of any possible uncertainty. The most widely used data qualifiers are:

J The analyte was positively identified; however, the result should be considered an estimated value.

UJ The analyte was not detected at or above the QL; however, the QL is considered an estimated value.

R QC parameters indicate the data are not usable.

Data with the data qualifier flag “R” will not be used in analysis of the analytical data. Data with limitations such as “J” and “UJ” may be used; however, the potential uncertainty in the results will be noted in future reports. Analytical data, to include data qualifier flags, will be uploaded to the Naval Installation Restoration Information Solution database using the Naval Electronic Data Deliverable format within 30 days of completion of validation.

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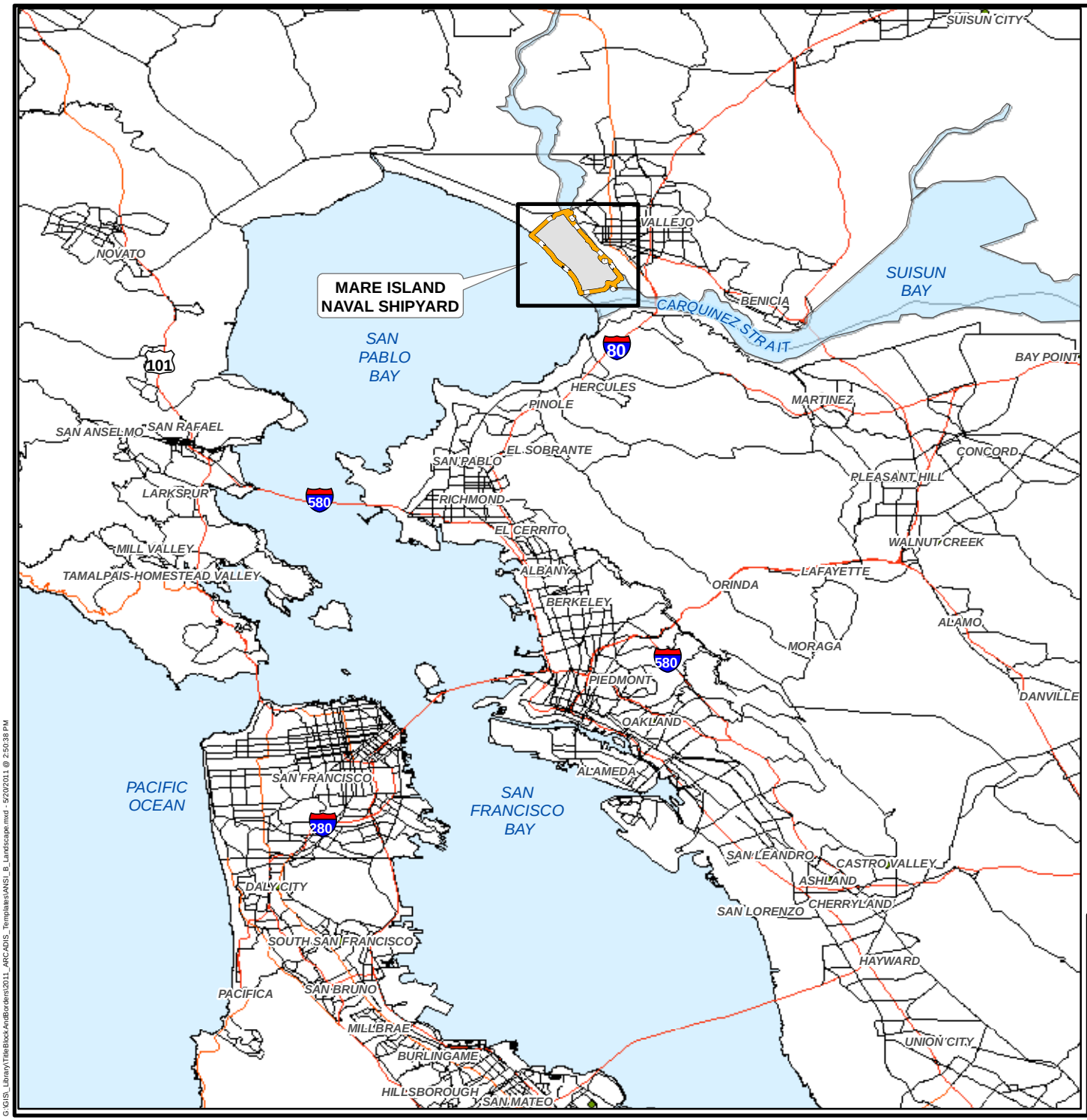
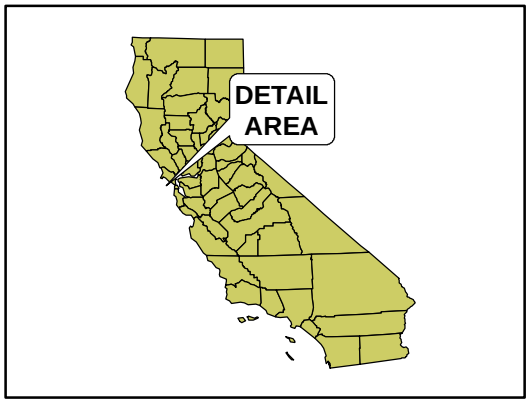
———. 2005. Environmental Work Instruction EVR.6 (EWI #6), Environmental Data Management and Required Electronic Delivery Standards. April.

FIGURES





LEGEND:

MARE ISLAND NAVAL SHIPYARD BOUNDARY

SOURCE: ENVIRONMENTAL SYSTEMS RESEARCH INSTITUTE (ESRI) USA SPATIAL FEATURE CLASSES

0 7 14
Miles

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

REGIONAL MAP

ARCADIS

FIGURE
1

CITY: SAN FRANCISCO DIV/GROUP: ENV/GIS DB: User: MEstifanos LD: PIC: PM: TM:
PROJECT: mavo0000/00000000/00000 PAT: 24 x 36
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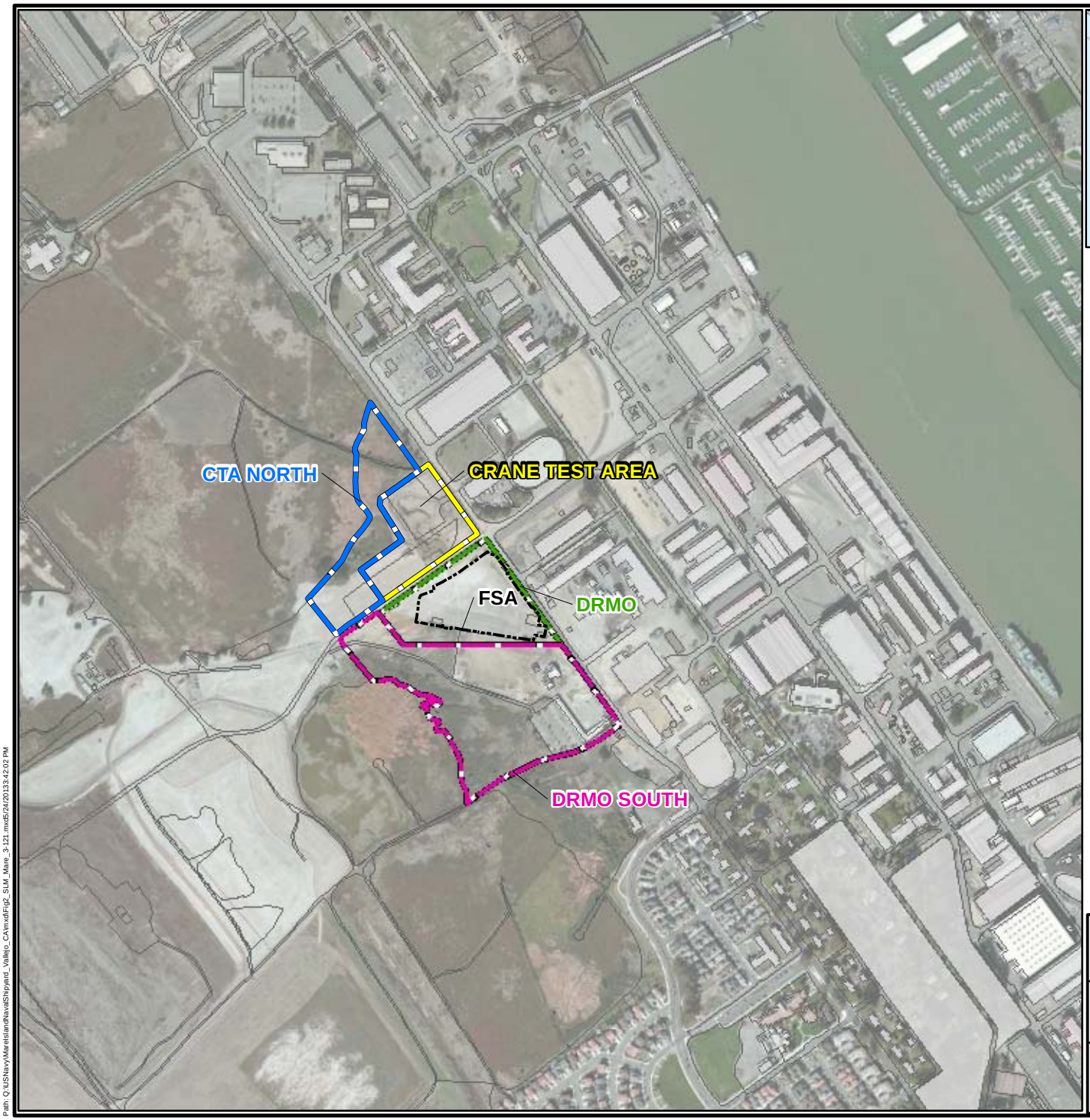
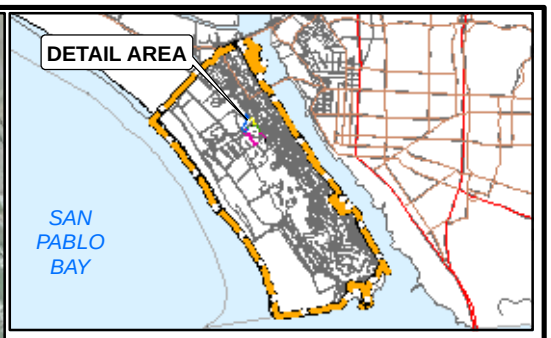




LEGEND:

- DRMO BOUNDARY
- DRMO SOUTH SITE BOUNDARY
- CTA NORTH SITE BOUNDARY
- CTA BOUNDARY (LENNAR MARE ISLAND)
- NAVY HISTORICAL BUILDING

ACRONYMS:
CTA - CRANE TEST AREA
DRMO - DEFENSE REUTILIZATION AND MARKETING OFFICE
FSA - FENCED SCRAPYARD AREA

SOURCE:
ENVIRONMENTAL SYSTEMS RESEARCH INSTITUTE (ESRI) USA SPATIAL FEATURE CLASSES. NAD83 STATE PLANE COORDINATE SYSTEM ZONE VI (FEET)
BASE MAP WAS OBTAINED FROM TREVET INC.
IMAGE © 2010 MICROSOFT CORPORATION AND ITS DATA SUPPLIERS, ACCESSED VIA ESRI ONLINE ON 3/26/13.

0 800 1,600
SCALE IN FEET

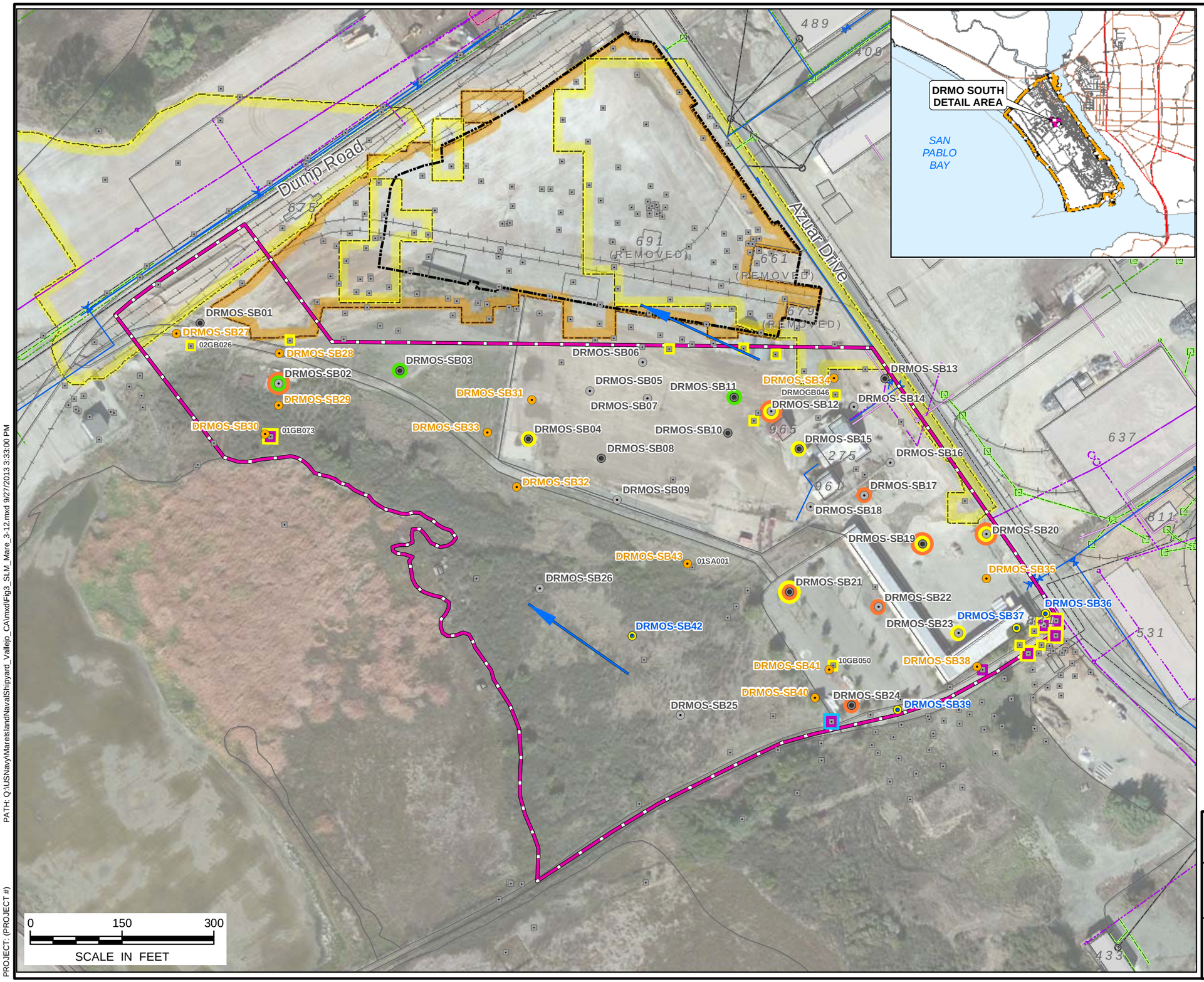
MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

SITE LOCATION MAP

ARCADIS

FIGURE
2

CITY: SAN FRANCISCO DIV/GROUP: ENV/GIS DB: User: MEstifanos LD: PIC: PM: TM:
PROJECT: FE0401 0000
Path: Q:\US\navy\Mareisland\NavalShipyard_Vallejo_CA\mxd\Fig2_SLM_Mare_3-121.mxd5/24/2013 3:42:02 PM



LEGEND

- SI SOIL SAMPLE LOCATION (2012)
- SI SOIL AND GROUNDWATER SAMPLE LOCATION (2012)
- PREVIOUSLY COLLECTED SAMPLE LOCATIONS
- PROPOSED SOIL/GROUNDWATER SAMPLING LOCATION
- PROPOSED SOIL SAMPLING LOCATION
- GROUNDWATER FLOW DIRECTION
- FENCED SCRAPYARD AREA
- DRMO SOUTH SITE BOUNDARY
- PCA EXCAVATION FOOTPRINT
- DRMO NTCRA BOUNDARY
- NAVY HISTORICAL BUILDING
- ELECTRICAL LINES
- SALTWATER PIPELINES
- GAS PIPELINES
- SANITARY SEWER PIPELINES
- WATER PIPELINES
- STORM DRAIN LINES
- INDUSTRIAL WASTE PIPELINES
- RAILROADS
- ARSENIC CONCENTRATION ABOVE SCREENING CRITERIA
- B(a)P CONCENTRATION ABOVE SCREENING CRITERIA
- DIESEL/MOTOR OIL CONCENTRATION ABOVE SCREENING CRITERIA
- LEAD CONCENTRATION ABOVE SCREENING CRITERIA
- AROCLOR 1260 CONCENTRATION ABOVE SCREENING CRITERIA

ACRONYMS:
B(a)P - BENZO(a)PYRENE
DRMO - DEFENSE REUTILIZATION AND MARKETING OFFICE
NTCRA - NON-TIME CRITICAL REMOVAL ACTION
PCA - PETROLEUM CORRECTIVE ACTION
RI - REMEDIAL INVESTIGATION
SI - SITE INSPECTION

SOURCE:
GOOGLE AERIAL IMAGE, DATE 8/4/2002
BASE MAP WAS OBTAINED FROM TREVET INC.

SOIL SCREENING CRITERIA ARE:
36 MILLIGRAMS PER KILOGRAM (mg/kg) FOR ARSENIC
0.21 mg/kg FOR B(a)P
100 mg/kg FOR DIESEL
500 mg/kg FOR MOTOR OIL
320 mg/kg FOR LEAD
1 mg/kg FOR AROCLOR 1260

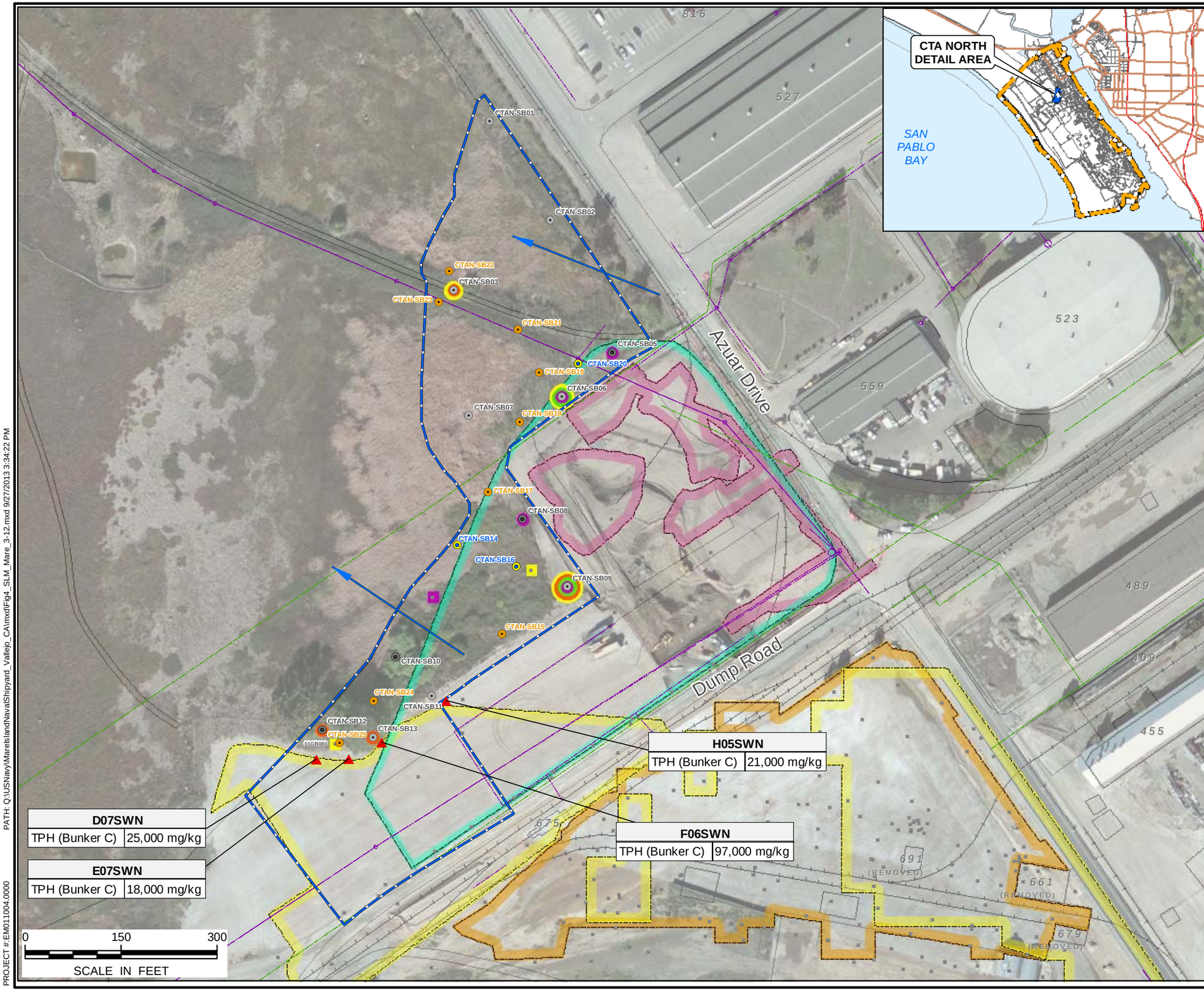
MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

DRMO SOUTH PROPOSED RI SAMPLING LOCATIONS

ARCADIS

FIGURE
3

CITY: SF DIV/GROUP: ENV/IM DB: MESTIFANOS LD: PIC: PM: TM: TR:
PROJECT: (PROJECT #) PATH: Q:\USNavy\MareIslandNavalShipyard_Vallejo_CA\mxd\Fig3_SLM_Mare_3-12.mxd 9/27/2013 3:33:00 PM



LEGEND

- SI SOIL SAMPLE LOCATION (2012)
- SI SOIL AND GROUNDWATER SAMPLE LOCATION (2012)
- PREVIOUSLY COLLECTED SAMPLE LOCATIONS
- SIDEWALL SAMPLE, CONCENTRATION ABOVE TPH CRITERION (PCA)
- GROUNDWATER FLOW DIRECTION
- ELECTRICAL UTILITY LINE
- SALTWATER UTILITY LINE
- CTA NORTH SITE BOUNDARY
- PCA EXCAVATION FOOTPRINT
- FILL AREA 1925-1942
- DRMO NTCRA BOUNDARY
- LMI CTA REMOVAL AREA FOOTPRINT
- NAVY HISTORICAL BUILDING
- RAILROADS
- PROPOSED SOIL/GROUNDWATER SAMPLING LOCATION
- PROPOSED SOIL SAMPLING LOCATION
- ARSENIC CONCENTRATION ABOVE SCREENING CRITERIA
- B(a)P CONCENTRATION ABOVE SCREENING CRITERIA
- LEAD CONCENTRATION ABOVE SCREENING CRITERIA
- DIESEL/MOTOR OIL CONCENTRATION ABOVE SCREENING CRITERIA

ACRONYMS:
B(a)P - BENZO(a)PYRENE
CTA - CRANE TEST AREA
LMI - LENNAR MARE ISLAND
NTCRA - NON-TIME CRITICAL REMOVAL ACTION
PCA - PETROLEUM CORRECTIVE ACTION
RI - REMEDIAL INVESTIGATION
SI - SITE INSPECTION
TPH - TOTAL PETROLEUM HYDROCARBONS

SOURCE:
GOOGLE AERIAL IMAGE, DATE 8/4/2002
BASE MAP WAS OBTAINED FROM TREVET INC.

SOIL SCREENING CRITERIA ARE:
36 MILLIGRAMS PER KILOGRAM (mg/kg) FOR ARSENIC
0.21 mg/kg FOR B(a)P
320 mg/kg FOR LEAD
100 mg/kg FOR DIESEL
500 mg/kg FOR MOTOR OIL

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

CTA NORTH PROPOSED RI SAMPLING LOCATIONS

ARCADIS

FIGURE
4

CITY: SF DIV/GROUP: ENV/IM DB: MESTIFANOS LD: PIC: PM: TM: TR:
PROJECT #:EM011004.0000 PATH: Q:\USNavy\MareIslandNavalShipyard_Vallejo_CA\mxd\Fig4_SLM_Mare_3-12.mxd 9/27/2013 3:34:22 PM

SOURCES/ RELEASE MECHANISMS	MIGRATION PATHWAYS	EXPOSURE MEDIA	EXPOSURE ROUTE	Potential Receptors				
				On-site Commercial/ Industrial Workers	On-site Utility and Construction Workers	Recreational Users	Residents (Hypothetical)	Ecological
Historical Releases to Soil or Groundwater from Former Activities	Volatilization	Air Vapors	Inhalation (Indoor Air)	--	--	--	--	--
			Inhalation (Outdoor Air)	○	○	○	○	--
	Leaching	Groundwater	Ingestion	--	--	--	--	○
			Dermal Contact	--	○	--	--	--
			Inhalation (from Potable Water)	--	--	--	--	--
	Wind Erosion and Transport	Dust Particles	Inhalation (Outdoor Air)	○	○	○	○	○
	Surface-Water Runoff Erosion and Transport	Surface and/or Subsurface Soil	Ingestion	○	○	○	○	○
			Dermal Contact	○	○	○	○	○
	Plant/Animal Uptake	Plant/Animal Tissue	Ingestion	--	--	--	--	○

○ Exposure pathway is complete or potentially complete;

-- Incomplete exposure pathway.

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

EXPOSURE PATHWAY SUMMARY

ARCADIS

FIGURE
5

ATTACHMENTS

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Attachment 1: Field Forms

ARCADIS

Daily Log

Project No.: _____ Page _____ of _____

Site Location: _____

Prepared By: _____

Date	Time	Description of Activities

ARCADIS

Document Control Number:TGM - _____

TGM + *project number plus date as follows: xxxxxxxx.xxxx.xxxxx - dd/mm/year*

TAILGATE HEALTH & SAFETY MEETING FORM

This form documents the tailgate meeting conducted in accordance with the Project HASP. Personnel who perform work operations on-site during the day are required to attend this meeting and to acknowledge their attendance, at least daily.

Project Name:			**Project Location:**
Date:	**Time:**	**Conducted by:**	**Signature/Title:**
Client:		**Client Contact:**	**Subcontractor companies:**

TRACKing the Tailgate Meeting

Think through the Tasks (list the tasks for the day):

1 _____ 3 _____ 5 _____

2 _____ 4 _____ 6 _____

Other Hazardous Activities - Check the box if there are any other ARCADIS, Client or other party activities that may pose hazards to ARCADIS operations ☐ If there are none, write "None" here: _____

If yes, describe them here: _____

How will they be controlled? _____

Prework Authorization - check activities to be conducted that require permit issuance or completion of a checklist or similar before work begins:

	Doc #		Doc #		Doc #
☐ Not applicable		☐ Working at Height		☐ Confined Space	
☐ Energy Isolation (LOTO)		☐ Excavation/Trenching		☐ Hot Work	
☐ Mechanical Lifting Ops		☐ Overhead & Buried Utilities		☐ Other permit	

Discuss following questions (for some review previous day's post activities). **Check if yes :**

		☐ Topics from Corp H&S to cover?
☐ Incidents from day before to review?	☐ Lessons learned from the day before?	☐ Any Stop Work Interventions yesterday?
☐ Any corrective actions from yesterday?	☐ Will any work deviate from plan?	☐ If deviations, notify PM & client
☐ JLAs or procedures are available?	☐ Field teams to "dirty" JLAs, as needed?	☐ All equipment checked & OK?
☐ Staff has appropriate PPE?	☐ Staff knows Emergency Plan (EAP)?	☐ Staff knows gathering points?

Comments: _____

Recognize the hazards (check all those that are discussed) (Examples are provided) and **Assess** the Risks (Low, Medium, High - circle risk level) - Provide an overall assessment of hazards to be encountered today and briefly list them under the hazard category.

☐ Gravity (i.e., ladder, scaffold, trips) (L M H)	☐ Motion (i.e., traffic, moving water) (L M H)	☐ Mechanical (i.e., augers, motors) (L M H)
☐ Electrical (i.e., utilities, lightning) (L M H)	☐ Pressure (i.e., gas cylinders, wells) (L M H)	☐ Environment (i.e., heat, cold, ice) (L M H)
☐ Chemical (i.e., fuel, acid, paint) (L M H)	☐ Biological (i.e., ticks, poison ivy) (L M H)	☐ Radiation (i.e., alpha, sun, laser) (L M H)
☐ Sound (i.e., machinery, generators) (L M H)	☐ Personal (i.e. alone, night, not fit) (L M H)	☐ Driving (i.e. car, ATV, boat, dozer) (L M H)

Continue TRACK Process on Page 2

TAILGATE HEALTH & SAFETY MEETING FORM - Pg. 2

Control the hazards (Check all and discuss those methods to control the hazards that will be implemented for the day): Review the HASP, applicable JLAs, and other control processes. Discuss and document any additional control processes.

☒ **STOP WORK AUTHORITY** (Must be addressed in every Tailgate meeting - (*See statements below*)

☐	Elimination	☐	Substitution	☐	Isolation
☐	Engineering controls	☐	Administrative controls	☐	Monitoring
☐	General PPE Usage	☐	Hearing Conservation	☐	Respiratory Protection
☐	Personal Hygiene	☐	Exposure Guidelines	☐	Decon Procedures
☐	Emergency Action Plan (EAP)	☐	Fall Protection	☐	Work Zones/Site Control
☐	JLA to be developed/used ***(specify)***	☐	LPO conducted ***(specify job/JLA)***	☐	Traffic Control
☐		☐		☐	Other ***(specify)***
☐		☐		☐	

Signature and Certification Section - Site Staff and Visitors

Name/Company/Signature	Initial & Sign in Time	Initial & Sign out Time	I have read and understand the HASP

Important Information and Numbers	Visitor Name/Co - not involved in work
All site staff should arrive fit for work. If not, they should report to the supervisor any restrictions or concerns. In the event of an injury, employees will call WorkCare at 1.800.455.6155 and then notify the field supervisor who will, in turn, notify Corp H&S at 1.720.344.3844. In the event of a motor vehicle accident, employees will notify the field supervisor who will then notify Corp H&S at 1.720.344.3844 and then Corp Legal at 1.720.344.3756. In the event of a utility strike or other damage to property of a client or 3rd party, employees will immediately notify the field supervisor, who will then immediately notify Corp Legal at 1.678.373.9556 and Corp H&S at 1.720.344.3500	In Out In Out In Out In Out

I will STOP the job any time anyone is concerned or uncertain about health & safety or if anyone identifies a hazard or additional mitigation not recorded in the site, project, job or task hazard assessment.

I will be alert to any changes in personnel, conditions at the work site or hazards not covered by the original hazard assessments.

If it is necessary to **STOP THE JOB,** I will perform **TRACK**; and then amend the hazard assessments or the HASP as needed.

I will not assist a subcontractor or other party with their work unless it is absolutely necessary and then only after I have done TRACK and I have thoroughly controlled the hazard.

Post Daily Activities Review - Review at end of day or before next day's work (Check those applicable and explain:)

☐ Lessons learned and best practices learned today: _____

☐ Incidents that occurred today: _____

☐ Any Stop Work interventions today? _____

☐ Corrective/Preventive Actions needed for future work: _____

☐ Any other H&S issues: _____

Keep H&S 1st in all things

WorkCare - 1.800.455.6155
Near Loss Hotline - 1.866.242.4304

ARCADIS

Real Time Air Monitoring Data Collection Forms

Document all air monitoring conducted on the Site below based on Section E of the HASP. Keep this form with the project files.

Site Name: _____ Date: _____

Instrument: _____ Model: _____ Serial #: _____

Calibration Method: (material used, settings, etc.)	
Calibration Results:	
Calibrated By:	

Activity Being Monitored	Compounds Monitored	Time	Reading	Action Required? Y/N

Describe Any Actions Taken as a Result of this Air Monitoring and Why:

_____

_____

_____

_____

_____

Signed: _____

Site Safety Officer

Make additional copies of this form as necessary.

ARCADIS
Infrastructure, environment, buildings

ID#:

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:

Contact & Company Name:	Telephone:
Address:	Fax:
City State Zip	E-mail Address:
Project Name/Location (City, State):	Project #:
Sampler's Printed Name:	Sampler's Signature:

Preservative							
Filtered (✓)							
# of Containers							
Container Information							

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: _____
G. Other: _____
H. Other: _____

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: _____
10. Other: _____

Matrix Key:
SO - Soil
W - Water
T - Tissue
SE - Sediment
SL - Sludge
A - Air
NL - NAPL/Oil
SW - Sample Wipe
Other: _____

Sample ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix								REMARKS

Special Instructions/Comments:

☐ **Special QA/QC Instructions(✓):**

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: _____	Date/Time:	Date/Time:	Date/Time:	Date/Time:

Distribution: WHITE – Laboratory returns with results YELLOW – Lab copy PINK – Retained by ARCADIS

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY	DATE TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

Oakland, CA • Houston, TX • Chicago, IL • Richmond, VA
(510) 465-4988 www.essvial.com (800) 233-8425

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY	DATE TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

Oakland, CA • Houston, TX • Chicago, IL • Richmond, VA
(510) 465-4988 www.essvial.com (800) 233-8425

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY

LOCATION

ANALYSIS

Oakland, CA • Houston, TX
(510) 465-4988 www.es

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY	DATE TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

Oakland, CA • Houston, TX • Chicago, IL • Richmond, VA
(510) 465-4988 www.essvial.com (800) 233-8425

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY	DATE TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

Oakland, CA • Houston, TX • Chicago, IL • Richmond, VA
(510) 465-4988 www.essvial.com (800) 233-8425

ENVIRONMENTAL SAMPLING SUPPLY

LOT#

SAMPLE ID

SAMPLED BY

LOCATION

ANALYSIS

Oakland, CA • Houston, TX
(510) 465-4988 www.es

CUSTODY SEAL

ENVIRONMENTAL SAMPLING SUPPLY

9601 San Leandro St. Oakland, CA 800-233-8425

Date: _____

Signature: _____

PEEL HERE

CUSTODY SEAL

ENVIRONMENTAL SAMPLING SUPPLY

9601 San Leandro St. Oakland, CA 800-233-8425

Date: _____

Signature: _____

PEEL HERE

CUSTODY SEAL

ENVIRONMENTAL SAMPLING SUPPLY

9601 San Leandro St. Oakland, CA 800-233-8425

Date: _____

Signature: _____

PEEL HERE

Utilities and Structures Checklist

Project: _____
Project Number: _____
Date: _____
Work locations applicable to this clearance checklist:

Pre-Field Work

One Call or "811" notified 48-72 hours in advance of work? ☐ Yes ☐ No
Utility companies notified during the One Call process ☐ See attached ticket

_____ _____ _____ _____
_____ _____ _____ _____
_____ _____ _____ _____

List any other utilities requiring notification: ☐ None

_____ _____ _____ _____

Client provided utility maps or "as built" drawings showing utilities? ☐ Yes ☐ No

Field Work

Markings present: ☐ Paint ☐ Pin flags/stakes ☐ Other ☐ None
Subsurface Utility Lines of Evidence Used (3 Minimum):

☐ One Call/"811"
☐ Client Provided Maps/Drawings
☐ Client Clearance
☐ Interviews: Name(s)/Affiliation(s) _____
_____
_____

Did persons interviewed indicate depths of any utilities in the subsurface?
☐ Yes, depths provided:

☐ Did not know or refused to answer

Comments:

☑ Site Inspection
☐ GPR
☐ Air-Knife
☐ Hydro-Knife
☐ Public Records/Maps
☐ Radiofrequency
☐ Metal Detector
☐ Handauger
☐ Potholing
☐ Probing
☐ Private Locator: Name and Company: _____
☐ Marine Locator: Name and Company: _____
☐ Other:

_____
_____

Tips for Successful Utility Location:
1. No excessive turning or downward force of handaugers/shovels, etc.
2. No hammering- no pickaxes-no digging bars-no hurrying or shortcutting
3. Select alternate/backup locations for clearance
4. Utilities may run directly under asphalt/concrete or be > 5 ft depth
5. Be on site when utilizing private utility locators

T R A C K

Site Inspection

During inspections look for the following ("YES" requires follow up investigation):

		Utility color codes		
a)	Natural gas line present (evidence of a gas meter)?	Yellow	☐ Yes	☐ No
b)	Evidence of subsurface electric lines :	Red		
	i) Conduits to ground from electric meter?		☐ Yes	☐ No
	ii) Overhead electric lines absent		☐ Yes	☐ No
	iii) Light poles, electric devices with no overhead lines?		☐ Yes	☐ No
c)	Evidence of water lines:	Blue		
	i) Water meter on site?		☐ Yes	☐ No
	ii) Fire hydrants in vicinity of work?		☐ Yes	☐ No
	iii) Irrigation systems?		☐ Yes	☐ No
d)	Evidence of sewers or storm drains:	Green		
	i) Restrooms or kitchen on site?		☐ Yes	☐ No
	ii) Gutter down spouts going into ground		☐ Yes	☐ No
	iii) Grates in ground in work area		☐ Yes	☐ No
e)	Evidence of telecommunication lines:	Orange		
	i) Fiber optic warning signs in areas?		☐ Yes	☐ No
	ii) Lines from cable boxes running into ground?		☐ Yes	☐ No
	iii) Conduits from power poles running into ground?		☐ Yes	☐ No
	iv) Aboveground boxes or housings in work area?		☐ Yes	☐ No
f)	Underground storage tanks:			
	i) Tank pit present?		☐ Yes	☐ No
	ii) Product lines running to dispensers/buildings?		☐ Yes	☐ No
	iii) Vent present away from tank pit?		☐ Yes	☐ No
g)	Proposed excavation markings in work area?	White	☐ Yes	☐ No
h)	Other:			
	i) Evidence of linear asphalt or concrete repair		☐ Yes	☐ No
	ii) Evidence of linear ground subsidence or change in vegetation?		☐ Yes	☐ No
	iii) Manholes or valve covers in work area?		☐ Yes	☐ No
	iv) Warning signs ("Call Before you Dig", etc) on or adjacent to site?		☐ Yes	☐ No
	v) Utility color markings not illustrated in this checklist?		☐ Yes	☐ No
i)	Aboveground lines in or near the work area:			
	i) < 50 kV within 10 ft of work area?		☐ Yes	☐ No
	ii) >50 - 200 kV within 15 ft of work area?		☐ Yes	☐ No
	iii) >200-350 kV within 20 ft of work area?		☐ Yes	☐ No
	iv) >350-500 kV within 25 ft of work area?		☐ Yes	☐ No
	v) >500-750 kV within 35 ft or work area?		☐ Yes	☐ No
	vi) >750-1000 kV within 45 ft of work area?		☐ Yes	☐ No

Comments:

Do not initiate intrusive work if utilities are suspected to be present in area and are not located, markings are over 14 days old, or if clearance methods provide incomplete or conflicting information. Do not perform intrusive work within 30 inches of a utility marking without hand clearing.

Name and signature of person completing the checklist:

Name: _____

Signature: _____

Date: _____

ARCADIS
Infrastructure, environment, buildings

Soil Boring Sample Log

Well/Boring _____ **Project Name and No.** _____

Site Location _____ **Drilling Started** _____ **Drilling Completed** _____

Total Depth Drilled _____ **feet** **Hole Diameter** _____ **inches** **Sampling Interval** _____ **feet**

Length and Diameter of Sampling Device _____ **Type of Sampling Device** _____

Drilling Method _____ **Drilling Fluid Used** _____

Drilling Contractor _____ **Driller** _____ **Helper** _____

Prepared By _____ **Hammer Weight** _____ **Hammer Drop** _____ **inches**

Sample Depth (feet below land surface) From	To	Sample Recovery (feet)	Time/Hydraulic Pressure or Blows per 6 inches	Sample Description	PID (ppm)

Soil Boring Sample Log (Continued)

Well/Boring _____ **Project Name and No.** _____

Prepared By _____

Sample Depth (feet below land surface) From	To	Sample Recovery (feet)	Time/Hydraulic Pressure or Blows per 6 inches	Sample Description	PID (ppm)

ARCADIS Infrastructure, environment, buildings	Boring ID: Permit No.:	**Project Name:** **Site Address:** **Project No.**

Drilling Co.		Logged By:	
Drilling Crew:			
Drilling Method:	Drill Bit and Size:	Borehole Diam:	
Total Depth Drilled (ft bgs):	Method of Completion:		
Begin Date:	Completion Depth:		
End Date:	▽ (ft bgs):	▼ (ft bgs):	Date:
Northing/Easting or Lat/Long:		Survey Grid System:	
Ground Surface Elev.:	Top of Casing Elev.:	Date Surveyed:	Surveyor:
Description of Boring Location:			

Well Design	Interval	Fmn Mat	Sand Pk/Int/Amt	Slot Size/Int	Trans. Mat/Int/Amt	Annul Seal/Mat/Int/Amt
Well Use:	GS-Int 1:	-----	-----	----		
Casing Material:	Int. 1:					
Casing Size:	Int. 2:					
Casing Schedule:	Int. 2:					
Max. Borehole Angle:		Surface Seal Comp:			Surface Completion:	

Depth	Blows per Foot or Pressure in psi	Drive Interval	Reccovery	Sample ID	Time	Headspace: gastech/PID/FID ppm	USCS Class	USCS Symbol	Well Const.	Description: Major Material, Density/Consistency, Color, Moisture, Additional Descriptions
0										

Notes:

Depth in feet below ground surface (bgs)

Registered Geologist Reviewing Logs:

ARCADIS
Infrastructure, environment, buildings

Boring ID:

Permit No.:

Project Name:
Site Address:
Project No.

Depth	Blows per Foot or Pressure in psi	Drive Interval	Reccovery	Sample ID	Time	Headspace: gastech/PID/FID ppm	USCS Class	USCS Symbol	Well Const.	Description: Major Material, Density/Consistency, Color, Moisture, Additional Descriptions

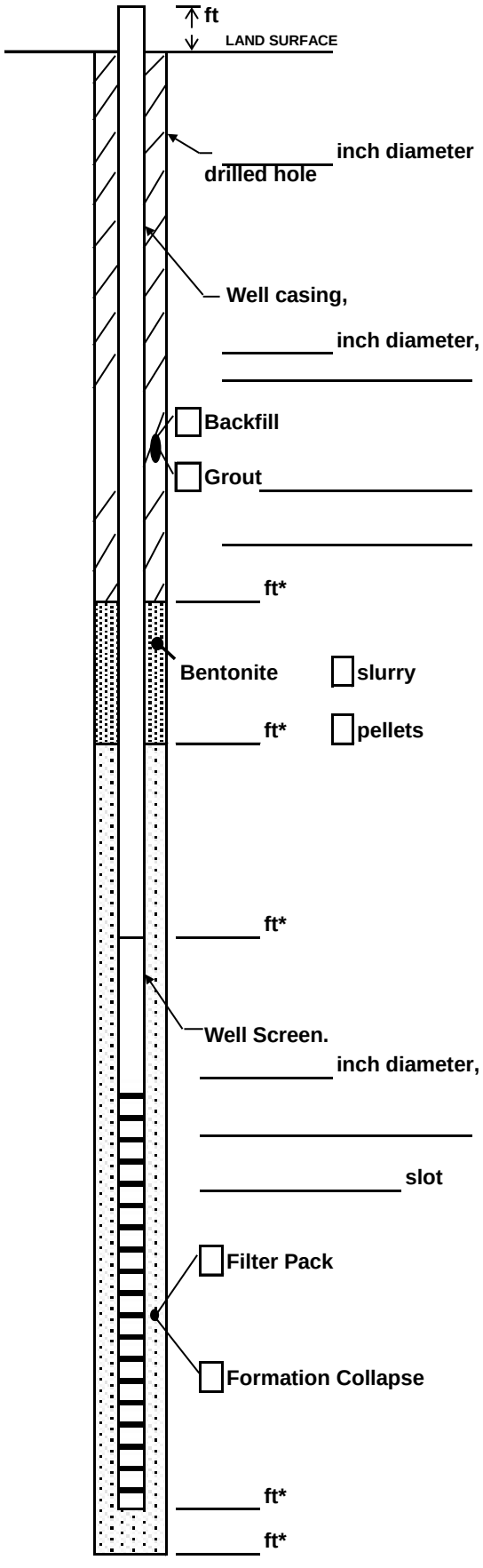
Notes:

Depth in feet below ground surface (bgs)

Registered Geologist Reviewing Logs:

ARCADIS
Infrastructure, environment, buildings

Well Construction Log
(Unconsolidated)



Project Name and No. _____

Well _____ Town/City _____

County _____ State _____

Permit No. _____

Land-Surface Elevation and Datum:

_____ feet ☐ Surveyed

☐ Estimated

Installation Date(s) _____

Drilling Method _____

Drilling Contractor _____

Drilling Fluid _____

Development Technique(s) and Date(s)

Fluid Loss During Drilling _____ gallons

Water Removed During Development _____ gallons

Static Depth to Water _____ feet below M.P.**

Pumping/Bailing Depth to Water _____ feet below M.P.**

Pumping/Bailing Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose _____

Remarks _____

* Depth Below Land Surface

**Measuring Point is Top of Well Casing Unless Otherwise Noted.

Prepared by _____

ARCADIS
Infrastructure, environment, buildings

Well Development Log

Project Number: _____ Well ID: _____
Date: _____ Developed By: _____
Weather: _____ Recorded By: _____

Instrument Identification

	PID	Water Quality Meter(s)
Model		
Serial #:		

Purging Information

Casing Material: _____ Development Technique: _____
Casing Diameter: _____ Screen Interval: From: _____ To: _____
Total Depth: _____
Depth to Water: _____ Volumes to be Purged: _____
Water Column: _____ Total Volume Purged: _____
Gallons/Foot: _____
Gallons in Well: _____

Well Casing Volumes (gal/ft):	2" = 0.16	3" = 0.37
	$3^1/_2$" = 0.50	4" = 0.65
	6" = 1.46	

Field Parameter Measurements Taken During Purging

Time	Volume Purged (gallons)	DTW (ft)	Temp ($^oF/^oC$)	pH (SI Units)	Turbidity (NTUs)	Spec Cond (µmhos/cm)	Comments / Observations

Remarks / Comments:

_____
_____
_____

Completed By: _____ Signature: _____
Reviewed By: _____ Date: _____

ARCADIS
Infrastructure, environment, buildings

Low-Flow Groundwater Sampling Form

Project Number: _____ Task: _____ Well ID: _____
Date: _____ Sampled By: _____
Weather: _____ Recorded By: _____
Coded Duplicate No.: _____

Instrument Identification

	PID	Water Quality Meter(s)
Model		
Serial #:		

Purging Information

Casing Material: _____
Casing Diameter: _____
Total Depth: _____
Depth to Water: _____
Water Column: _____
Gallons/Foot: _____
Gallons in Well: _____

Purge Technique (circle one): Low-Flow Remove 3 Well Volumes Bail Dry
Purge Equipment (circle one): Submersible Centrifugal Bladder Peristaltic Bailer
Screen Interval: From: _____ To: _____
Pump Intake Setting: _____
Volumes to be Purged: _____
Total Volume Purged: _____
Pump On: _____ Off: _____

Well Casing Volumes (gal/ft):	2" = 0.16	3" = 0.37
	$3^1/_2$" = 0.50	4" = 0.65
	6" = 1.46	

Field Parameter Measurements Taken During Purging

Time	Minutes Elapsed	Flow Rate ( )	Volume Purged ( )	DTW (ft btoc)	Turbidity (NTUs)	ORP (mV)	pH (SI Units)	Spec Cond (µmhos/cm)	Temp (ºC)	DO (mg/L)	Comments
Criteria:		<500 mL/min	N/A	drawdown <0.3 ft	±10%	±10 mV	±0.1 units	±3%	±3%	±10%	

Observations During Sampling
Well Condition: _____ Purge Water Disposal: _____
Color: _____ Turbidity (qualitative): _____
Odor: _____ Other: _____

Sample ID: _____ Sample Date & Time: _____
Samples Analyzed For: _____

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Attachment 2: Laboratory Certification

The American Association for Laboratory Accreditation

World Class Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

CURTIS & TOMPKINS, LLC

Berkeley, 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 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the current 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 10th day of January 2012.

President & CEO
For the Accreditation Council
Certificate Number 2943.01
Valid to February 28, 2014

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

CURTIS & TOMPKINS, LLC.[1]
2323 5th Street
Berkeley, CA, 94710
Carolyn Brizzolara Phone: (510) 204 2237
carolyn.brizzolara@ctberk.com

ENVIRONMENTAL

Valid To: February 28, 2014 Certificate Number: 2943.01

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

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), IR Spectrometry, Titrimetry, Total Organic Carbon

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Metals						
Aluminum	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Antimony	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Arsenic	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Barium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Beryllium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B

5301 Buckeystown Pike, Suite 350 | Frederick, Maryland 21704-8373 | Phone: 301 644 3248 | Fax: 301 662 2974 | www.A2LA.org

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Cadmium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Calcium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Chromium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Cobalt	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Copper	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Iron	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Lead	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Magnesium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Manganese	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Mercury	- - - - -	- - - - -	EPA 7470A	EPA 7470A	EPA 7470A / 7471A / 7471B	EPA 7470A / 7471A / 7471B
Molybdenum	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Nickel	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Potassium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Selenium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Silver	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Sodium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Thallium	- - - - -	- - - - -	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Vanadium	-----	-----	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Zinc	-----	-----	EPA 6010B / 6010C / 6020 / 6020A	EPA 200.8 / 3010A	EPA 6010B / 6010C / 6020 6020A	EPA 3050B
Nutrients						
Ammonia (as N)	-----	-----	SM 4500NH3-D	SM 4500NH3-D	SM 4500NH3-D	SM 4500NH3-D
Kjeldahl Nitrogen	-----	-----	SM 4500NH3-C	SM 4500NH3-C	SM 4500NH3-C	SM 4500NH3-C
Nitrate (as N)	-----	-----	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056
Nitrite (as N)	-----	-----	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056
Perchlorate	-----	-----	EPA 314.0	EPA 314.0	-----	-----
Total phosphorus	-----	-----	SM 4500P-E	SM 4500P-E	-----	-----
Demands						
Biochemical Oxygen Demand	-----	-----	SM 5210B	SM 5210B	-----	-----
Chemical Oxygen Demand	-----	-----	SM 5220D	SM 5220D	-----	-----
Total Organic Carbon	-----	-----	SM 5310C	SM 5310C	-----	-----
Wet Chemistry						
Alkalinity	SM 2320B	SM 2320B	SM 2320B	SM 2320B	-----	-----
Bromide	-----	-----	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056
Chloride	-----	-----	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056
Cyanide	-----	-----	SM 4500 CN-E / EPA 9010B / 9014	SM 4500 CN-E / EPA 9010B / 9014	SM 4500 CN-E / EPA 9010B / 9014	SM 4500 CN-E / EPA 9010B / 9014
Amenable Cyanide	-----	-----	SM 4500 CN-E / EPA 9010B / 9014	SM 4500 CN-E / EPA 9010B / 9014	EPA 9010B / 9014	EPA 9010B / 9014
Ferrous Iron	-----	-----	SM 3500Fe-B	SM 3500Fe-B	-----	-----
Flash Point	-----	-----	EPA 1010	EPA 1010	-----	-----
Fluoride	-----	-----	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056
Hexavalent Chromium	-----	-----	EPA 7196A / 7199 / SM 3500Cr-B,C	EPA 7196A / 7199 / SM 3500Cr-B,C	EPA 3060 / 7196A	EPA 3060 / 7196A
pH	-----	-----	EPA 9040B / SM 4500-H^+ B	EPA 9040B / SM 4500-H^+ B	EPA 9045C	EPA 9045C
Specific Conductance	-----	-----	SM 2510 B	SM 2510 B	-----	-----

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Sulfate	- - - - -	- - - - -	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056	EPA 300.0 / 9056
Sulfide	- - - - -	- - - - -	SM 4500S2-D	SM 4500S2-D	SM 9034	SM 9030B
Total Dissolved Solids	- - - - -	- - - - -	SM 2540C	SM 254C	- - - - -	- - - - -
Total Suspended Solids	- - - - -	- - - - -	SM 2540D	SM 2540 D	- - - - -	- - - - -
Purgeable Organics (volatiles)						
Gas Range Organics (GRO)	- - - - -	- - - - -	EPA 8015B / 8015D	EPA 5030B / 5030C	EPA 8015B / 8015D	EPA 5030B / 5030C / 5035 / 5035A
Acetone	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Benzene	- - - - -	- - - - -	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C / 5035 / 5035A
Bromobenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Bromochloromethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Bromodichloromethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Bromoform	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Bromomethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
2-Butanone	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Tert-Butyl Alcohol (TBA)	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
n-Buytlbenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
sec-Butylbenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
tert-Butylbenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Carbon Disulfide	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Carbon Tetrachloride	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Chlorobenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Chloroethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Chloroform	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Chloromethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
2-Chlorotoluene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
4-Chlorotoluene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Dibromochloromethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,2-Dibromo-3-chloropropane (DBCP)	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Dibromomethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,2 Dibromomethane (EDB)	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,2-Dichlorobenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,3-Dichlorobenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,4-Dichlorobenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Dichlorodifluoromethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,1-Dichloroethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,2-Dichloroethane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
1,1-Dichloroethene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
cis-1,2-Dichloroethene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
trans-1,2-Dichloroethene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,2-Dichloropropane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,3-Dichloropropane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
2,2-Dichloropropane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,1-Dichloropropene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
cis-1,3-Dichloropropene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
trans-1,3-Dichloropropene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Ethyl Benzene	-----	-----	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C / 5035 / 5035A
Ethyl tert-Butyl Ether (ETBE)	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
2-Hexanone	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Hexachlorobutadiene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Isopropylbenzene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Isopropyl Ether (DIPE)	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Para-Isopropyltoluene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Methylene Chloride	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
4-Methyl-2-pentanone	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Methyl tert-amyl Ether (TAME)	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Methyl tert-butyl ether (MTBE)	-----	-----	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C / 5035 / 5035A
Naphthalene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
n-Propylbenzene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Styrene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,1,1,2-Tetrachloroethane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,1,2,2-Tetrachloroethane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Tetrachloroethene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Toluene	-----	-----	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C / 5035 / 5035A
1,2,3-Trichlorobenzene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,2,4-Trichlorobenzene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,1,1-Trichloroethane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,1,2-Trichloroethane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Trichloroethene	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Trichlorofluoromethane	-----	-----	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
1,2,3-Trichloropropane	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,2,4-Trimethylbenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
1,3,5-Trimethylbenzene	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Vinyl acetate	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
Vinyl chloride	- - - - -	- - - - -	EPA 8260B / 8260C	EPA 5030B / 5030C	EPA 8260B / 8260C	EPA 5030B / 5030C / 5035 / 5035A
m,p-Xylene	- - - - -	- - - - -	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C / 5035 / 5035A
o-Xylene	- - - - -	- - - - -	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C	EPA 8260B / 8260C / 8021B	EPA 5030B / 5030C / 5035 / 5035A
Extractable Organics (semivolatiles)						
DRO	- - - - -	- - - - -	EPA 8015B / 8015D	EPA 3520C	EPA 8015B / 8015C / 8015D/	EPA 3550B / 3550C / 3546
Acenaphthene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Acenaphthylene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Anthracene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Benzoic Acid	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270 C 8270D	EPA 3550B / 3550C
Benzo (a) Anthracene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Benzo (b) Fluoranthene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Benzo (k) Fluoranthene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Benzo (ghi) Fluoranthene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Benzo (a) Pyrene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 / 8270 C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Benzyl Alcohol	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Bis (2-chloroethoxy) Methane	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Bis (2-chloroethyl) Ether	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Bis (2-chloroisopropyl) Ether	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Bis (2-ethylhexyl) Phthalate	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
4-Bromophenyl-Phenylether	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Butyl Benzyl Phthalate	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
4-Chloroaniline	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
4-Chloro-3-methylphenol	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2-Chloronaphthalene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
2-Chlorophenol	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
4-Chlorophenyl Phenyl Ether	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Chrysene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Dibenzo (a,h) Anthracene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Dibenzofuran	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
1,2-Dichlorobenzene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
1,3-Dichlorobenzene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
1,4-Dichlorobenzene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
3,3'-Dichlorobenzidine	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2,4-Dichlorophenol	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Diethyl Phthalate	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2,4-Dimethylphenol	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Dimethyl Phthalate	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Di-n-butyl Phthalate	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Di-n-octyl Phthalate	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
4,6-Dinitro-2-methylphenol	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2,4-Dinitrophenol	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2,4-Dinitrotoluene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2,6-Dinitrotoluene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
1,4 - Dioxane			EPA 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
1,2-Diphenylhydrazine reported as Azobenzene due to breakdown	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Fluoroanthene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Fluorene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Hexachlorobenzene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Hexachlorobutadiene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Hexachloro cyclopentadiene	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Hexachloroethane	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Indeno (1,2,3-cd) pyrene	- - - - -	- - - - -	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
Isophorone	- - - - -	- - - - -	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
1-Methylnaphthalene	- - - - -	- - - - -	EPA 8270C-SIM / 8270D-DIM	EPA 3520C	EPA 8270C-SIM / 8270D-DIM	EPA 3550B / 3550C

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
2-Methylnaphthalene	-----	-----	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
2-Methylphenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
4-Methylphenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Naphthalene	-----	-----	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
2-Nitroaniline	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
3-Nitroaniline	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
4-Nitroaniline	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Nitrobenzene	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2-Nitrophenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
4-Nitrophenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
N-Nitrosodi-n-propylamine	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
N-Nitrosodimethylamine	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
N-Nitrosodiphenylamine	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Pentachlorophenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Phenanthrene	-----	-----	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Phenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Pyrene	-----	-----	EPA 8310 / 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3520C	EPA 8310 8270C / 8270D / 8270C-SIM / 8270D-SIM	EPA 3550B / 3550C
1,2,4-Trichlorobenzene	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2,4,5-Trichlorophenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
2,4,6-Trichlorophenol	-----	-----	EPA 8270C / 8270D	EPA 3520C	EPA 8270C / 8270D	EPA 3550B / 3550C
Pesticides/PCBs						
Aldrin	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
alpha-BHC	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
beta-BHC	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
delta-BHC	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
gamma-BHC	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Chlordane (technical)	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
alpha-Chlordane	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
gamma-Chlordane	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
4,4'-DDD	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
4,4'-DDE	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
4,4',-DDT	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Dieldrin	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Endosulfan I	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Endosulfan II	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Endonsulfan Sulfate	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Endrin	-----	-----	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Endrin Aldehyde	- - - - -	- - - - -	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Endrin Ketone	- - - - -	- - - - -	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Heptachlor	- - - - -	- - - - -	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Heptachlor Epoxide	- - - - -	- - - - -	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Methoxychlor	- - - - -	- - - - -	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
PCB-1016 (Arochlor)	- - - - -	- - - - -	EPA 8082 / 8082A	EPA 3520C	EPA 8082 / 8082A	EPA 3550B / 3550C
PCB-1221	- - - - -	- - - - -	EPA 8082 / 8082A	EPA 3520C	EPA 8082 / 8082A	EPA 3550B / 3550C
PCB-1232	- - - - -	- - - - -	EPA 8082 / 8082A	EPA 3520C	EPA 8082 / 8082A	EPA 3550B / 3550C
PCB-1242	- - - - -	- - - - -	EPA 8082 / 8082A	EPA 3520C	EPA 8082 / 8082A	EPA 3550B / 3550C
PCB-1248	- - - - -	- - - - -	EPA 8082 / 8082A	EPA 3520C	EPA 8082 / 8082A	EPA 3550B / 3550C
PCB-1254	- - - - -	- - - - -	EPA 8082 / 8082A	EPA 3520C	EPA 8082 / 8082A	EPA 3550B / 3550C
PCB-1260	- - - - -	- - - - -	EPA 8082 / 8082A	EPA 3520C	EPA 8082 / 8082A	EPA 3550B / 3550C
Toxaphene	- - - - -	- - - - -	EPA 8081A / 8081B	EPA 3520C	EPA 8081A / 8081B	EPA 3550B / 3550C
Nitroaromatics & Nitramines						
2-Amino-4,6-dinitrotoluene	- - - - -	- - - - -	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
4-Amino-2,6-dinitrotoluene	- - - - -	- - - - -	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
3,5-Dinitroaniline	- - - - -	- - - - -	----------	----------	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
1,3-Dinitrotoluene	- - - - -	- - - - -	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
2,4-Dinitrotoluene	- - - - -	- - - - -	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
2,6-Dinitrotoluene	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
HMX	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
Nitroglycerine	-----	-----	----------	----------	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
Nitrobenzene	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
2-Nitrotoluene	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
3-Nitrotoluene	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
4-Nitrotoluene	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
Pentaerythritol (PETN)	-----	-----	----------	----------	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
RDX	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
Tetryl	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
1,3,5-Trinitrobenzene	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
2,4,6-Trinitrotoluene	-----	-----	EPA 8330 / 8330A	EPA 3535	EPA 8330 / 8330A / 8330A Modified	EPA 8330 / 8330A
Hazardous Waste Characteristics						

Parameter/Analyte	Potable Water	Potable Water Prep Methods	Nonpotable Water	Nonpotable Water Prep Methods	Solid Hazardous Waste	Solid Hazardous Waste Prep Methods
Synthetic Precipitation Leaching Procedure (SPLP)	-----	-----	-----	-----	EPA 1312	EPA1312
Toxicity Characteristic Leaching Procedure (TCLP)	-----	-----	-----	-----	EPA 1311	EPA 1311
Air						
Carbon Dioxide	-----	-----	RSK-175	RSK-175	-----	-----
Ethane	-----	-----	RSK-175	RSK-175		
Ethene	-----	-----	RSK-175	RSK-175	-----	-----
Methane	-----	-----	RSK-175	RSK-175	-----	-----
Acetylene	-----	-----	RSK-175	RSK-175	-----	-----

Parameter/Analyte	Air Analysis
1,1,1-Trichloroethane	TO-15
1,1,2,2-Tetrachloroethane	TO-15
1,1,2-Trichloroethane	TO-15
1,1-Dichloroethane	TO-15
1,1-Dichloroethene	TO-15
1,2,4-Trichlorobenzene	TO-15
1,2,4-Trimethylbenzene	TO-15
1,2-Dibromoethane	TO-15
1,2-Dichlorobenzene	TO-15
1,2-Dichloroethane	TO-15
1,2-Dichloropropane	TO-15
1,3,5-Trimethylbenzene	TO-15
1,3-Butadiene	TO-15
1,3-Dichlorobenzene	TO-15
1,4-Dichlorobenzene	TO-15
2-Butanone	TO-15
2-Hexanone	TO-15
4-Ethyltoluene	TO-15
4-Methyl-2-Pentanone	TO-15
Acetone	TO-15
Acrolein	TO-15
Benzene	TO-15
Benzyl chloride	TO-15
Bromodichloromethane	TO-15
Bromoform	TO-15
Bromomethane	TO-15
Carbon Disulfide	TO-15
Carbon Tetrachloride	TO-15
Chlorobenzene	TO-15
Chloroethane	TO-15
Chloroform	TO-15
Chloromethane	TO-15
Cyclohexane	TO-15

Parameter/Analyte	Air Analysis
Dibromochloromethane	TO-15
Ethyl Acetate	TO-15
Ethylbenzene	TO-15
Freon 113	TO-15
Freon 114	TO-15
Freon 12	TO-15
Hexachlorobutadiene	TO-15
MTBE	TO-15
Methylene Chloride	TO-15
Napthalene	TO-15
Propylene	TO-15
Styrene	TO-15
Tetrachloroethene	TO-15
Tetrahydrofuran	TO-15
Toluene	TO-15
Trichloroethene	TO-15
Trichlorofluoromethane	TO-15
Vinyl Acetate	TO-15
Vinyl Chloride	TO-15
cis-1,2-Dichloroethene	TO-15
cis-1,3-Dichloropropene	TO-15
m,p-Xylenes	TO-15
n-Heptane	TO-15
n-Hexane	TO-15
o-Xylene	TO-15
trans-1,2-Dichloroethene	TO-15
trans-1,3-Dichloropropene	TO-15

Gamma Spectroscopy Parameter/Analyte	Solid Hazardous Waste
Actinium – 228	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Americium – 241	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Bismuth – 212	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Bismuth – 214	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Cesium – 137	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Cobalt – 60	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Europium – 152	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Europium – 154	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Lead – 210	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Lead – 212	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Lead – 214	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Protactinium – 234M	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Radium – 226	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Thallium - 208	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Thorium – 232	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Uranium - 235	Modified EPA Method 901.1/DoE HASL 300 Ga-01

1. **This accreditation covers testing performed at the main laboratory listed above and the following satellite laboratory listed below for the following tests:**

201A & 201B Fischer Ave
Hunters Point Naval Ship Yard
San Francisco, CA 94124

Gamma Spectroscopy Parameter/Analyte	Solid/Wipe Analysis
Actinium – 228	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Americium – 241	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Bismuth – 212	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Bismuth – 214	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Cesium – 137	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Cobalt – 60	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Europium – 152	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Europium – 154	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Lead – 210	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Lead – 212	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Lead – 214	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Potassium – 40	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Protactinium – 234M	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Radium – 226	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Thallium - 208	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Thorium – 232	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Thorium – 234	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Uranium - 235	Modified EPA Method 901.1/DoE HASL 300 Ga-01
Gross Alpha Beta Spectroscopy Gas Proportional Counter	
Alpha & Beta Radiation	Modified EPA Method 9310

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA LABORATORIES WEST SACRAMENTO
880 Riverside Parkway
West Sacramento, CA 95605
Douglas Weir Phone: 916 3744 389

ENVIRONMENTAL

Valid To: January 31, 2014 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	**Non potable Water**	**Solid Hazardous Waste**	**Air**	**Associated Prep Methods**
Metals				
Aluminum	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Antimony	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Arsenic	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Barium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Beryllium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Cadmium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Calcium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Chromium (Total)	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Chromium (Hexavalent)	EPA 7196A	EPA 7196A	-------	EPA 3005A/3010A/3050A
Cobalt	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Copper	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Iron	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Lead	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Magnesium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A

5301 Buckeystown Pike, Suite 350 | Frederick, Maryland 21704-8373 | Phone: 301 644 3248 | Fax: 301 662 2974 | www.A2LA.org

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Manganese	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Mercury	EPA 7470A	EPA 7471A	-------	EPA 3005A/3010A/3050A
Molybdenum	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Nickel	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Potassium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Selenium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Silver	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Sodium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Thallium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Vanadium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Zinc	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Nutrients				
Nitrate	EPA 353.2/9056A/300.0	EPA 353.2/9056A/300.0	-------	----------
Nitrate-nitrite	EPA 353.2/ SM4500-NO3 F	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	-------	----------
Wet Chemistry				
Oil and Grease	EPA 1664A/9070	EPA 1664A/9071	-------	----------
Nitrocellulose	WS-WC-0050	WS-WC-0050	-------	----------
Perchlorate	EPA 6850	EPA 6850	-------	----------
Chloride	EPA 9056A/300.0	EPA 9056A/300.0	-------	----------
Fluoride	EPA 9056A/300.0	EPA 9056A/300.0	-------	----------
Sulfate	EPA 9056A/300.0	EPA 9056A/300.0	-------	----------
			-------	----------
Hazardous Waste Characteristics				
TCLP Extractables	----------	EPA 1311		
TCLP Inorganics	----------	EPA 1311	-------	----------
			-------	----------
Purgeable Organics				
(volatiles)				
Acetone	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Acrolein	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Acrylonitrile	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Allyl Chloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Benzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromodichloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromoform	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromomethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Carbon disulfide	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Carbon tetrachloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Chloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2-Chloroethyl vinyl ether	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chloroform	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chloroprene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Cyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1-Chlorocyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Dibromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dibromo-3-chloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dibromoethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Dibromomethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,3-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,4-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
T-1,4-Dichloro-2-Butene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Dichlorodifluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
trans-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
cis-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,3-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
cis-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
trans-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,4-Dioxane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Ethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Ethylmethacrylate	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Hexachlorobutadiene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Hexane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2-Hexanone (MBK)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Iodomethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Isobutanol	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Isopropyl Ether	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methacrylonitrile	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methylene chloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methyl ethyl ketone	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methyl Methacrylate	EPA 8260B/8260C	EPA 8260B/8260C	-------	----------
4-Methyl-2-pentanone (MIBK)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Naphthalene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Propionitrile	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,1,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,2,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Tetrachloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Tetrahydrofuran	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Toluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,3-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,4-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,1-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,2-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-2-Trichloro-1,2-2-trifluorethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Trichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Trichlorofluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,3-Trichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Vinyl acetate	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Vinyl chloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
m & p xylene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
o-xylene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Xylenes, Total	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
tert-amyl methyl ether (TAME)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
tert-butyl alcohol (TBA)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Ethyl tert-butyl ether (ETBE)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
n-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
sec-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
tert-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
4-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Isopropylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
n-Propylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Styrene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,4-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,3,5-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Oxygenates	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Gasoline Range Organics (GRO)	EPA 8260B/AK101	EPA 8260B/AK101	-------	EPA 5030A/5030B/5035/5035A
p-Isopropyltoluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Extractable Organics				
(semivolatiles)				
Acenaphthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Acenaphthylene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Acetophenone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Acetylaminofluorene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Aminobiphenyl	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Aniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Anthracene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
7,12-Dimethylbenz(a)anthracene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Aramite	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzaldehyde	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Dibenze(a,j)acridine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzo(a)anthracene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(b)fluoranthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(k)fluoranthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(j)fluoranthene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzo(g,h,i)perylene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(a)pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(e)pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzoic acid	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzyl alcohol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzyl butyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-sec-Butyl-4,6-dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Bis(2-chloroethoxy) Methane	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Bis(2-chloroethyl) ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Bis(2-chloroisopropyl) ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Di(2-ethylhexyl) phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Bromophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Carbazole	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
4-Chloroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Chloro-3-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexacloropropene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Chlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Chlorophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Chrysene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Diallate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3510C/3550B/ 3580A
Dibenz(a,h)anthracene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Dibenzofuran	EPA 8270C/8270D	EPA 8270C/8270D	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
1,2-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,3-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,4-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3,3'-Dichlorobenzidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,6-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Diethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dimethylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Dimethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Di-n-butyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Di-n-octyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4,6-Dinitro-2-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,6-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,4-Dioxane	WS-MS-0011	WS-MS-0011	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
1,2-Diphenylhydrazine (as Azobenzene)	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Famphur	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Fluoranthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Fluorene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Hexachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexachlorobutadiene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexachlorocyclopentadiene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexachloroethane	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Indeno(1,2,3-c,d)pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Isodrin	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isophorone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isosafrole	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isosafrole #1	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isosafrole #2	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Kepone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Dimethoate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Methapyrilene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Methyl methanesulfonate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Methyl Parathion	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3-Methylcholanthrene	EPA 8270C/8270D	EPA 8270C/8270D		EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Methylnaphthalene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
2-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3&4-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Naphthalene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
1,4-Naphthoquinone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1-Methylnaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1-Naphthylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Naphthylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Nitro-o-toluidine-1-oxide	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
5-Nitro-o-toluidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Nitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,2-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,3-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,4-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,3,5-Trinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodimethylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodi-n-propylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodiphenylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodiethylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitroso-di-n-butylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosomethylethylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosomorpholine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosopyrrolidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosopiperidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Parathion	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
p-Chorobenzilate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
p-Dimthylaminoazobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentaclorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentacloronitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentacloroethane	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phenacetin	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phenanthrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
a,a-Dimethylphenethlamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Biphenyl	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Diphenylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
p-Phenylenediamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Picoline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phorate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Promamide	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Pyridine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Safrole	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Sulfotepp	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Disulfotone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Ethylmethanesulfonate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Thionazin	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,2,4,5-Tetrachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,2,4-Trichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
2,4,5-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,3,4,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,3,5,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4,6-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Diesel Range Organics (DRO)	EPA 8015B/8015C/AK102	EPA 8015B/8015C/AK102	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Residual Range Organics	AK103	AK103	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
o,o,o-TEPT	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
o-Toluidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
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	-------	----------
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	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
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	-------	----------
1,4-Oxathiane	WS-MS-0003	WS-MS-0003	-------	----------
Dimethyl Disulfide	WS-MS-0003	WS-MS-0003	-------	----------
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	-------	----------
Ethylene glycol dinitrate	EPA 8330B	EPA 8330B	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
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	-------	----------
Nitroguanidine	WS-LC-0010	WS-LC-0010	-------	----------
Nitrosamines				
N-Nitrosodimethylamine (NDMA)	WS-MS-0012	WS-MS-0012	-------	----------
Perfluoro Compounds				
Perfluorooctanoic acid	WS-LC-0020	WS-LC-0020	-------	----------
Perfluorooctane sulfonate	WS-LC-0020	WS-LC-0020	-------	----------
Pesticides/PCBs				
Aldrin	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
a-BHC	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
b-BHC	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
d-BHC	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
g-BHC (Lindane)	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
a-Chlordane	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
g-Chlordane	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Oxy-Chlordane	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
4,4'-DDD	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
2,4'-DDD	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
4,4'-DDE	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
2,4'-DDE	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
4,4'-DDT	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
2,4'-DDT	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Dieldrin	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endosulfan I	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endosulfan II	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endosulfan sulfate	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endrin	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endrin aldehyde	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endrin ketone	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Heptachlor	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Heptachlor epoxide	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Hexachlorobenzene	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Methoxychlor	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Cis-Nonachlor	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Trans-Nonachlor	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Toxaphene	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Mirex	EPA 1699	EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Chlordane (technical)	EPA 8081A/8081B	EPA 8081A/8081B	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
<u>PCB (Aroclors)</u>				
PCB-1016	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A/ 3620B/3665A
PCB-1221	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/
PCB-1232	EPA 8082/8082A	EPA 8082/8082A	-------	3550B/3550C/3620B/3660A/
PCB-1242	EPA 8082/8082A	EPA 8082/8082A	-------	3620B/3665A
PCB-1248	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/
PCB-1254	EPA 8082/8082A	EPA 8082/8082A	-------	3550B/3550C/3620B/3660A/
PCB-1260	EPA 8082/8082A	EPA 8082/8082A	-------	3620B/3665A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB-1262	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/
PCB-1268	EPA 8082/8082A	EPA 8082/8082A	-------	3550B/3550C/3620B/3660A/
PCB (congeners)				
PCB 1 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 2 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 3 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 4 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 5 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 6 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 7 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 8 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 9 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 10 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 11 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 12 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 13 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 14 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 15 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 16 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 17 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 18 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 19 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 20 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 21 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 22 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 23 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 24 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 25 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 26 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 27 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 28 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 29 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 30 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 32 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 31 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 33 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 34 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 35 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 36 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 37 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 38 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 39 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 40 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 41 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 42 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 43 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 44 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 45 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 46 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 47 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 48 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 49 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 50 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 51 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 52 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 53 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 54 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 55 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 56 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 57 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 58 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 59 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 60 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 61 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 62 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 63 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 64 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 65 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 66 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 67 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 68 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 69 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 70 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 71 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 72 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 73 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 74 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 75 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 76 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 77 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 78 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 79 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 80 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 81 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 82 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 83 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 84 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 85 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 86 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 87 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 88 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 89 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 90 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 91 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 92 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 93 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 94 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 95 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 96 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 97 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 98 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 99 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 100 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 101 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 102 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 103 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 104 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 105 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 106 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 108 (BZ)/107 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 109 (BZ)/108 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 107 (BZ)/109 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 110 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 111 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 112 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 113 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 114 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 115 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 116 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 117 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 118 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 119 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 120 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 121 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 122 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 123 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 124 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 125 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 126 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 127 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 128 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 129 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 130 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 131 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 132 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 133 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 134 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 135 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 136 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 137 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 138 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 139 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 140 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 141 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 142 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 143 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 144 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 145 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 146 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 147 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 148 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 149 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 150 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 151 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 152 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 153 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 154 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 155 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 156 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 157 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 158 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 159 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 160 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 161 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 162 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 163 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 164 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 165 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 166 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 167 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 168 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 169 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 170 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 171 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 172 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 173 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 174 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 175 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 176 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 177 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 178 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 179 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 180 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 181 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 182 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 183 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 184 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 185 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 186 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 187 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 188 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 189 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 190 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 191 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 192 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 193 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 194 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 195 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 196 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 197 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 198 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 201 (BZ)/199 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 199 (BZ)/200 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 200 (BZ)/201 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 202 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 203 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 204 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 205 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 206 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 207 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 208 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 209 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
Hormones,Steroids, Pharmaceuticals and Disinfection Byproducts				
Acetominophen	WS-LC-0024	----------	-------	----------
Atenolol	WS-LC-0024	----------	-------	----------
Azithromycin	WS-LC-0024	----------		
Carbadox	WS-LC-0024	----------	-------	----------
Carbamazepine	WS-LC-0024	----------	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Clarithromycin	WS-LC-0024	----------		
Diazepam	WS-LC-0024	----------	-------	----------
Digoxigenin	WS-LC-0024	----------	-------	----------
Digoxin	WS-LC-0024	----------		
Diphenylhydramine	WS-LC-0024	----------	-------	----------
Fluoxetine	WS-LC-0024	----------		
Flumequine	WS-LC-0024	----------	-------	----------
Gemfibrozil	WS-LC-0024	----------		
Ibuprofen	WS-LC-0024	----------	-------	----------
Naproxen	WS-LC-0024	----------	-------	----------
Ormetoprim	WS-LC-0024	----------	-------	----------
Penicillin G	WS-LC-0024	----------		
Sulfachloropyridazine	WS-LC-0024	----------	-------	----------
Sulfadiazine	WS-LC-0024	----------	-------	----------
Sulfamethizole	WS-LC-0024	----------	-------	----------
Sulfamethoxazole	WS-LC-0024	----------	-------	----------
Sulfathiazole	WS-LC-0024	----------	-------	----------
Thiabendazole	WS-LC-0024	----------		
Trimethoprim	WS-LC-0024	----------		
Tris (2-chloro-2-propyl)phosphate (TCPP)	WS-LC-0024	----------	-------	----------
Warfarin	WS-LC-0024	----------	-------	----------

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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*).

AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
CORPORATE SEAL 1978
DISTRICT OF COLUMBIA
A2LA

Presented this 27th day of March 2012.

President & CEO
For the Accreditation Council
Certificate Number 2928.01
Valid to January 31, 2014
Revised June 1, 2012

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

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Attachment 3: Laboratory Standard Operating Procedures
(See attached Compact Disc)

Curtis & Tompkins, Ltd

Polynuclear Aromatic Hydrocarbons (PAH) and 1,4-Dioxane by EPA 8270-SIM

Approved by:	Signature:	Date:
Cheryl Pacheco Operations Manager	Cheryl Bowden	2/22/13
Teresa Morrison QA Director	Teresa Morrison	2/15/13
Reapproved:		

This document contains information that may only be disseminated to C&T staff, clients & regulators.

Polynuclear Aromatic Hydrocarbons (PAH) and 1,4-Dioxane by EPA 8270-SIM

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This document contains information that may only be disseminated to C&T staff, clients & regulators.

Polynuclear Aromatic Hydrocarbons (PAH) and 1,4-Dioxane by EPA 8270-SIM

1.0 SCOPE

This analysis is only to be performed by analysts who are familiar with, and fully qualified to perform, the normal 8270 analysis of methylene chloride extracts of soil and water samples, as this document is only a summary of the critical volumes and steps.

Methylene chloride extracts are analyzed by a Selected Ion Monitoring (SIM) procedure based on EPA 8270. Using SIM, the detector collects information for *only* those ions expected in the target analytes and does not do a full scan. Library searches cannot be performed on the resulting data and background contaminants, particularly heavy hydrocarbons, can readily contribute false positives and/or a high bias; see the "*Discussion*" section below for further details.

See Appendix_3 for target compounds and reporting limits.

Discussion

A common misconception is that 8270-SIM is a method guaranteed to achieve positive compound identification with much lower reporting limits for the PAH's than either normal 8270 or the HPLC method 8310. For relatively clean samples this is often true, however the data user should be aware that there are significant matrix interferences that may contribute to both higher reporting limits and false positive results.

In GC-MS methods, the detector bombards the compounds with electrons as the compounds exit the GC column. This splits the target compounds into ions that have mass:charge abundances in characteristic ratios. If there are enough other types of molecules, various versions of alkanes (not just straight chain) for example, other ions may also be produced.

For the GC/MS "Scan Mode", which is used for a normal 625 or 8270 analysis, the detector scans linearly across every ion mass in the range (35 – 450 m/z). This mode allows compounds to be identified by pattern recognition and quantified by the abundance of the characteristic ions.

For the GC/MS "SIM mode", which is the acronym for "Selected Ion Monitoring", the detector scans only for a limited number of ions, thus spending more time at each mass allowing the detector to pick up ions occurring at lower abundances (concentrations); more time equals a greater chance to gather a significant and quantifiable signal.

The limitation of the SIM method is that, by looking only for specific ions, the pattern-recognition capabilities of the GC-MS are limited to determining that those ions characteristic of the target compounds are either present at quantifiable levels or are not present. If the ions are present, the target compound must be quantified and reported the

hit; there is no information on other masses that would allow assessment of any contributions from background hydrocarbons or other non-target interferents.

2.0 REFERENCES

Analysis Method:
EPA 8000B, *Determinative Chromatographic Separations*, SW-846, Dec 1996
EPA 8000C, *Determinative Chromatographic Separations*, SW-846, March 2003
EPA 8270C, Semivolatile Organics by GC/MS, SW-846, Update 3, Dec.1996
EPA 8270D, Semivolatile Organics by GC/MS, SW-846, Revision 4, February 2007

Extraction Methods:
EPA 3500B, Organic Extraction and Sample Preparation, SW-846, Update 3, Dec.1996
EPA 3510C, Separatory Funnel Liquid-Liquid Extraction, SW-846 Update 3, Dec 1996
EPA 3520C, Continuous Liquid-Liquid Extraction, SW-846, Update 3, Dec.1996
EPA 3550B, Sonication Extraction, SW-846 Update 3, Dec. 1996
EPA 3580A, Waste Dilution, SW-846 Update 1, July 1992

Optional Cleanup Method:
EPA 3640A, Gel-Permeation Cleanup, SW-846, Update 3, Dec.1996

Other References:
NELAC Standards, June 2003
Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Rev 4.2 October 2010
C&T SOP QA 1.5, *Calibrating & Maintaining Temperature Controls*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limit (MDL) & Limit of Detection (LOD)*
C&T SOP QA 4.6, *Establishing the Limit of Quantitation (LOQ)*
C&T SOP QA 9.6, *Insuring Compliant Manual Integration*
Operator Manuals for HP5890, Agilent 6890, and Agilent 6892 GC's
Operator Manuals for HP5971, HP5972, HP5973, and Agilent 5975 MSD's
Manuals for "Chemstation" software and operating system

3.0 PRESERVATION & HOLDING TIMES

Water:	Preservation:	No chemical preservative. Store at >0.0 to 6ºC.
	Holding Times:	7 days from collection until extraction; 40 days from extraction until analysis.
Soil:	Preservation:	Store at 4°C.
	Holding Time:	14 days from collection until extraction; 40 days from extraction until analysis.

4.0 SAFETY

Assume all standards, samples, and extracts may contain hazardous and/ or potentially toxic chemicals.

5.0 QC REQUIREMENTS & PROCEDURE

The calibration, batch QC, and daily sequence requirements and acceptance criteria are essentially the same as for the full-scan 8270 analysis. A Performance Evaluation Mix (PEM) is generally not required because this analysis does not include thermally labile compounds.

Note: For DoD QSM project sample analyses, a PEM is required, unless a variance has been approved by the client

The QC Requirements are summarized below. See the full BNA SOP (SVOC 8.1) for full discussion and details of the daily procedure, including running initial calibrations, daily sequences, and using the software/LIMS.

5.1 Initial Tune & Calibration:

- DFTPP tune standard precedes curve & meets SW-846 8270 m/z ratio requirements
- Curve consists of at least 5 points bracketing the quantitation range.
- If using quadratic regression/ equation must use at least 6 points.
- For projects requiring 8270C, each compound must meet a minimum relative response factor of 0.05. For projects requiring 8270D, the RRF must meet the minimums listed in Appendix 9
- %RSD $\leq$ 30% for all compounds before using a curve
- Average response factor must be $\leq$ 15% or linear/ quadratic regression must be used.
- Linear or Quadratic regression Coefficient of Determination (COD or r^2) must be $\geq$ 0.99

5.2 Initial Calibration Verification (ICV):

An ICV, obtained from a second manufacturer, follows the ICAL and precedes samples, to demonstrate that the curve is valid.

EPA 8270C requires $\leq$ 20%D for the CCCs listed in Table 4 of the method but does not include limits for the other compounds; the CCCs are Acenaphthene, Fluoranthene, and Benzo(a)pyrene. For the non-CCC compounds, C&T uses a 30%D limit for non-DoD projects requiring 8270C.

Although DoD projects usually require 8270D, and 8270D Section 9.3 allows a 30%D acceptance window (recoveries between 70-130%), the DoD QSM 4.2 Table F-4 requires 20%D for all compounds.

5.3 Daily Tune & Continuing Calibration Verification (CCV):

- DFTPP tune standard run at beginning of each 12-hour tune-shift & SW-846 8270 m/z ratio requirements
- CCV run at beginning of each 12-hour tune shift, following a DFTPP tune standard.
- CCV levels varied within calibration range, not just at the mid-point
- For all projects requiring 8270C, the Relative Response Factor must be $\geq$ 0.05.
  Note: For all projects requiring EPA 8270D, the minimum relative response factors listed in Appendix 9 must be met.

- %D ≤ 20% for the CCCs (Acenaphthene, Benzo(a)pyrene, Fluoranthene) and ≤30% for others.
  ***Note**:* For all DoD QSM projects or any projects requiring EPA 8270D, the %D ≤ 20% for all target analytes. No more than 20% of the full compound list may fail %D, all compounds must meet minimum response.
- Retention times of the internal standards must be within 0.5 minutes of the RTs seen in the midpoint of the initial calibration.
- Internal standard response must be 50% to 200% of that seen in the midpoint of the initial calibration.
- Sufficient resolution between isomeric pairs must be demonstrated to report the isomers individually. Sufficient resolution between two peaks is achieved if the height of the valley between the two peaks is less than 50% of the average of the two peak heights.

5.4 **Internal Standards (same as full 8270):**
Added to every standard, sample, and batch QC sample:

1,4-Dichlorobenzene-d4	Naphthalene-d8
Acenaphthene-d10	Phenanthrene-d10
Chrysene-d12	Perylene-d12.

Retention time of the internal standards must be + 0.5 minutes from the CCV.
Area of each internal standard must fall between 50-200% of the midpoint of the ICAL.

5.5 **Batch QC:**
Method blank, laboratory control sample (LCS), matrix spike (MS) and matrix spike duplicate (MSD) is extracted and analyzed with each batch. If insufficient sample volume was submitted to extract an MS/MSD, a blank spike (BS) and blank spike duplicate (BSD) is extracted in place of the LCS/MS/MSD.

Method blank must be "ND" at the reporting limit and the LCS or BS/BSD must meet acceptance criteria. If either of these criteria is not met, corrective action must be taken. Control limits generated semi-annually, based on control charts of the previous year's data. See the associated SOP Table "8270-SIM QC Limits" for current in-house limits.

5.6 **Qualitative Analysis:**
See Appendix 6 for the ions monitored for each compound and the associated internal standards. Target analyte retention time and ion-ratio criteria are the same as for full 8270.

All surrogates must pass control chart limits unless obvious matrix interference is present. If interference is visible in the chromatogram surrogate failure can be reported without further analysis. If matrix is not clearly biasing the surrogate recovery corrective action must be taken.

For samples with high level hits a clean instrument blank, or equivalent, must be analyzed to insure against carryover before subsequent runs in the sequence can be reported. If an instrument blank is not run, but the next sample is ND to the MDL for the saturating

compound(s) it may serve as an "instrument blank" and the following samples in the sequence can be reported.

See the full BNA SOP (SVOC 8.1) for further details.

6.0 INTERFERENCES

The primary interference with this analysis comes from a high hydrocarbon background. See the Discussion section above for further details.

7.0 EQUIPMENT

All GC/MS systems used for this analysis are HP/Agilent instruments. Chemstation and C&T LIMS software systems are used for data acquisition and reduction. See the full BNA SOP (SVOC 8.1) for further details.

8.0 DAILY PROCEDURE

See the full BNA SOP (SVOC 8.1) for details on setting up, running, and working up the daily instrument sequence.

Note: A PEM must be included only if the sequence will contain DoD QSM samples for which a variance has not been received from the client.

9.0 POLLUTION PREVENTION

The split vent and septum purge lines are directed through a carbon trap in order to reduce solvent emissions into the laboratory.

10.0 WASTE DISPOSAL

Sample extracts should be kept in the freezer at < -10°C for at least 40 days after extraction. The extracts, and any expired standards, should then be transferred to the lab's "Chlorinated Solvents" waste stream for proper disposal.

11.0 REVISION HISTORY

Revision 4 was changed as follows:

- Cover page and numbering were reformatted
- Added Safety, Interferences, Equipment, Daily Procedure Sections, adding links to full BNA SOP
- Section 2: Updated to include EPA 3510C, EPA 8000B/8000C, NELAC 2003, C&T SOPs
- Section 3: Updated to match refrigeration requirements from NELAP/QSM of >0ºC to 6ºC
- Section 5: Corrected CCV
- Appendix_2: Updated for current source standards and working standards prep
- Appendix_3: Updated 1,4-Dioxane RL in Soil from 50 ug/Kg to 33 ug/Kg
- Appendix_5: Minor changes to reflect current settings

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APPENDIX_1: CALCULATIONS

Sample quantitation and batch QC results are calculated in the same manner as for the full-scan 8270 analysis. See the full BNA SOP (SVOC 8.1) for details.

APPENDIX_2: SOURCE & WORKING STANDARDS

SOURCE STANDARDS

Source standards are those purchased from a chemical manufacturer or vendor. For source standards, the LIMS S-name is unique to both the composition (compound list) of the standard *and* to the vendor of that standard. A new S-name must be assigned whenever the composition is changed or when the standard is obtained from a different vendor; the information must then be entered in the "Standard Definitions" table before the new standard is assigned an S#. If you need more details, log into the LIMS browser; follow the 'LAB MENU' link and click on the "New Standards System (March 2005)" link for details on the system.

Certificates of Analysis should be obtained from the vendor of each source standard; the certificates should be labeled with the LIMS ID and the date received and filed in the 3-ring binder. Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is one year from date received. Label each vial with the contents, LIMS S#, and expiration date.

When standard ampules are opened and used, excess volume should be transferred to a 2mL screw-cap vial and retained for no more than six months. Store all standards in the standards freezer between -10 and -20°C; do not store standards in the same freezer as samples or extracts.

Source Standards

Analytes	Concentration (ug/mL)	Supplier & Catalog#	LIMS SS Name
PEM / DFTPP	1,000	Restek # 31615	31615
Internal Standards Mix	4,000	Restek # 31006	SVOC IS
Primary Std: PAH/Surrogate 1,4-Dioxane	 200 1,000	Restek # 565927	CSTMSIM-R
PAH + Surrogates	200	Supelco # 21348078	CSTMSIMICV-
1,4-Dioxane	2,000	Supelco # 4-8367	4-8367

Alternate Source Standards

Analytes	Concentration (ug/mL)	Supplier & Catalog#	LIMS SS Name
PAH + Surrogates	200	Accustd # S-14764-R2	S-14764-R2
1,4-Dioxane	100	Accustd # APP-9-096	APP-9-096

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WORKING STANDARDS PREPARATION

Working standards are those prepared at C&T, which should be prepared in Class-A volumetric flasks. For working standards, the LIMS S-name is not unique to the source standard vendor but *is* unique to the compound list and concentrations contained in the working standard; if the concentration or compounds in the working standard changes, a new S-name, compound list and concentrations must be entered in the "Standard Definitions" table before the standard is logged in and assigned an S#. It is *very important* to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

The benchbook entry should include:

- prep date and initials of the analyst making the standard,
- LIMS S#, concentration, and volume of each Source Standard used,
- solvent name, volume, and lot#, and
- final volume, concentration, and expiration date of the new Working Standard.

Label the standards vials with the LIMS S# and the expiration date.

Working standards expire 180 days after preparation from the source standards unless any of the source standards expire before the 180 days. If any of the source standards expire before the 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards. **The expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**. Aliquots are taken from the standard vial daily and this aliquot is used for only one day before being discarded. Store all working standards in a freezer between -10 and -20°C; do not store standards in a freezer containing samples or extracts.

Expired standards may be used for trouble-shooting or method development so long as each standard vial is clearly marked "expired" and stored in a well-marked tray containing only expired standards.

Note: The "Final Volumes" listed below do not include the internal standards, as those are added proportionally to both standards and samples.

Standard & Conc. (µg/mL)	Final Volume (mL) in $MeCl_2$	Using Source Std	Add Vol (µL) Source Std	LIMS S- Name
PEM / DFTPP 50µg/mL	20.	31615	1,000	PEM/DFTPP
		MeCl2	19,000	
Internal Std 100µg/mL	5.0	SVOC IS	125	IS100PPM
		MeCl2	4,875	
ICV: PAH 1 µg/mL	5.0	CUSTMSIMICV-	25.	SIMICVB
+ 1,4-Dioxane 10 µg/mL		4-8367	25.	
+ Surrogates 1 µg/mL		IS100PPM	50.	
		MeCl2	4,950	

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8270-SIM
Initial Calibration Standards

ICAL Standard & Conc. (µg/mL)	Final Volume (mL)	Using Std	Add Vol (µL) Source Std	LIMS S- Name
ICAL 0.1 / 0.5 / 1.0	2.0	CSTMSIM-R	1.0	SIMICAL-R1
		IS100PPM	20.0	
		MeCl2	1,999	
ICAL 0.2 / 1.0 / 1.0	2.0	CSTMSIM-R	2.0	SIMICAL-R2
		IS100PPM	20.0	
		MeCl2	1998	
ICAL 0.5 / 2.5 / 1.0	10.0	CSTMSIM-R	25.0	SIMICAL-R3
		IS100PPM	100.	
		MeCl2	9,975	
ICAL 1.0 / 5.0 / 1.0	10.0	CSTMSIM-R	50	SIMICAL-R4
		IS100PPM	100	
		MeCl2	9,950	
ICAL 2.0 / 10. / 1.0	10.0	CSTMSIM-R	100	SIMICAL-R5
		IS100PPM	100	
		MeCl2	9,900	
ICAL 5.0 / 25. / 1.0	2.0	CSTMSIM-R	50.	SIMICAL-R6
		IS100PPM	20.	
		MeCl2	1,950	
ICAL 10 / 50 / 1.0	2.0	CSTMSIM-R	100	SIMICAL-R7
		IS100PPM	20	
		MeCl2	1,950	

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APPENDIX_3: **8270-SIM TARGET COMPOUNDS**
With Standard C&T Reporting Limits

Polynuclear Aromatic Hydrocarbons (PAH)
EPA 8270-SIM

CAS #	Compound	Water RL µg/L	Soil RL µg/Kg
83-32-9	Acenaphthene	0.1	5
208-96-8	Acenaphthylene	0.1	5
120-12-7	Anthracene	0.1	5
56-55-3	Benzo(a)anthracene	0.1	5
50-32-8	Benzo(a)pyrene	0.1	5
205-99-2	Benzo(b)fluoranthene	0.1	5
207-08-9	Benzo(k)fluoranthene	0.1	5
191-24-2	Benzo(g,h,i)perylene	0.1	5
218-01-9	Chrysene	0.1	5
53-70-3	Dibenz(a,h)anthracene	0.1	5
206-44-0	Fluoranthene	0.1	5
86-73-7	Fluorene	0.1	5
193-39-5	Indeno(1,2,3-cd)pyrene	0.1	5
91-20-3	Naphthalene	0.1	5
85-01-8	Phenanthrene	0.1	5
129-00-0	Pyrene	0.1	5
Additional Compounds (may be added to the compound list):			
90-12-0	1-Methylnaphthalene	0.1	5
91-57-6	2-Methylnaphthalene	0.1	5
Surrogates:			
321-60-8	2-Fluorobiphenyl		
4165-60-0	Nitrobenzene-d5		
1718-51-0	Terphenyl-d14		

1,4-Dioxane by EPA 8270-SIM

CAS #	Compound	Water RL µg/L	Soil RL µg/Kg
123-91-1	1,4-Dioxane	1	33
Surrogates:			
321-60-8	2-Fluorobiphenyl		
4165-60-0	Nitrobenzene-d5		
1718-51-0	Terphenyl-d14		

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APPENDIX_4: DILUTIONS

Dilutions should be prepared so that the concentration of the highest target analyte falls in the upper half of the calibration curve.

Let the extracts warm to room temperature then:

If a 2x dilution is required, dilute a 100µL aliquot of extract with 100µL methylene chloride in an insert, add 2uL of internal standard solution, and seal the vial with a crimp cap.

If the required dilution is >2x, dilute an aliquot of the extract to a total volume of 500uL in an autosampler vial, add 5µL internal standard solution and seal the vial with a crimp cap. See table below for appropriate volumes.

Shake the dilution and invert 3 times to mix.

Dilution Factor	Extract Volume (µL)	$MeCl_2$ Volume (µL)
2	100	100
5	100	400
10	50	450
20	25	475
50	10	490
100	5	495
200	2.5	497.5
250	2	498
500	1	499
1,000	1	999

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APPENDIX_5: INSTRUMENT CONDITIONS

The parameters listed below were current at the time this document was written and may change at the analyst's discretion to optimize instrument performance. Changes are documented in the instrument maintenance log.

Column: Restek RXI-5silMS (30m x 250 µm x 0.5 µm)
Injection Volume: 1 uL

Temperature Program:

Injector Temperature:	270 ºC
Detector Temperature:	300 ºC
Oven Temperature Program:	
Initial	40 ºC for 3.0 min
Ramp 1	23 ºC/min to 100 ºC
Ramp 2	16 ºC/min to 124 ºC
Ramp 3	19 ºC/min to 340 ºC
Final Time	4.00 min

Injector Flow:

Mode:	Pulsed Splitless
Pressure:	16.17 psi (on)
Pulse pressure:	30.0 psi
Pulse time:	0.70 min
Purge flow:	30.0 min
Purge time	0.69 min
Total flow:	34.6 min

Column Flow:

Mode:	constant flow
Initial Flow:	2.0 mL/min
Initial Time:	0 min
Initial Pressure:	16.18 psi

Detector:

Solvent Delay:	3.66 min
MS Quad:	150 °C
MS Source:	230 °C

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APPENDIX_6: MONITORED-ION GROUPING

The "Group Start Time" listed below is based on the June 2003 instrument parameters for BNA-03 and may be slightly different for other instruments. All other parameters should be the same across instruments.

SIM Group 1:

Resolution:	Low		
Group Start Time:	0 min		
Plot1 Ion:	88.00		
Ions/ Dwell (in group):	Mass	88.00	
	msec	100	

SIM Group 2:

Resolution:	Low			
Group Start Time:	3.67 min			
Plot1 Ion:	57.10			
Ions/ Dwell (in group):	Mass	57.1	58.1	88.0
	msec	100	100	100

SIM Group 3:

Resolution:	Low		
Group Start Time:	6.0 min		
Plot1 Ion:	115.0		
Ions/ Dwell (in group):	Mass	115.0	151.9
	msec	100	100

SIM Group 4:

Resolution:	Low							
Group Start Time:	8.0 min							
Plot1 Ion:	54.1							
Ions/ Dwell (in group):	Mass	54.1	68.1	82.1	127.0	128.0	127.0	136.0
	msec	15	15	15	15	15	15	15

SIM Group 5:

Resolution:	Low				
Group Start Time:	9.8 min				
Plot1 Ion:	142.0				
Ions/ Dwell (in group):	Mass	141.0	142.0	171.0	172.1
	msec	50	50	50	50

SIM Group 6:

Resolution:	Low							
Group Start Time:	11.0 min							
Plot1 Ion:	152.0							
Ions/ Dwell (in group):	Mass	151.0	152.0	153.0	154.0	160.1	162.1	164.1
	msec	15	15	15	15	15	15	15

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SIM Group 7:

Resolution:	Low						
Group Start Time:	12.0 min						
Plot1 Ion:	165.0						
Ions/ Dwell (in group):	Mass	165.0	166.0	167.0			
	msec	50	50	50			

SIM Group 8:

Resolution:	Low						
Group Start Time:	13.0 min						
Plot1 Ion:	178.0						
Ions/ Dwell (in group):	Mass	80.1	94.1	176.0	178.0	179.0	188.1
	msec	15	15	15	15	15	15

SIM Group 9:

Resolution:	Low							
Group Start Time:	14.5 min							
Plot1 Ion:	202.0							
Ions/ Dwell (in group):	Mass	101.0	122.1	200.0	202.0	203.0	212.1	244.2
	msec	15	15	15	15	15	15	15

SIM Group 10:

Resolution:	Low						
Group Start Time:	16.4 min						
Plot1 Ion:	228.0						
Ions/ Dwell (in group):	Mass	120.1	226.0	228.0	229.0	236.1	240.1
	msec	15	15	15	15	15	15

SIM Group 11:

Resolution:	Low			
Group Start Time:	17.8 min			
Plot1 Ion:	252.0			
Ions/ Dwell (in group):	Mass	125.0	252.0	253.0
	msec	50	50	50

SIM Group 12:

Resolution:	Low						
Group Start Time:	18.43 min						
Plot1 Ion:	252.0						
Ions/ Dwell (in group):	Mass	125.0	252.0	253.0	260.1	264.1	265.1
	msec	15	15	15	15	15	15

SIM Group 13:

Resolution:	Low							
Group Start Time:	19.3 min							
Plot1 Ion:	276.1							
Ions/ Dwell (in group):	Mass	138.0	139.0	227.0	276.1	277.1	278.1	279.0
	msec	15	15	15	15	15	15	15

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APPENDIX _7: MONITORED IONS & INTERNAL STANDARD ASSIGNMENT

Internal Standard	Related Compounds
1.) 1,4-Dichlorobenzene-d_4	compounds 1-2
3.) Naphthalene-d_8	compounds 3-7
8.) Acenaphthene-d_{10}	compounds 8-12
13.) Phenanthrene-d_{10}	compounds 13-16
17.) Chrysene-d_{12}	compounds 17-21
22.) Perylene-d_{12}	compounds 22-28

Compound	CAS Number	Primary Ion	Secondary Ion(s)	
1.) * 1,4-Dichlorobenzene-d_4	3855-82-1	152	150	115
2.) 1,4-Dioxane	123-91-1			
3.) * Naphthalene-d8	1146-65-2	136	68	
4.) $ Nitrobenzene-d5	4165-60-0	82	128	54
5.) Naphthalene	91-20-3	128	129	127
6.) 2-Methylnaphthalene	91-57-6	142	141	
7.) 1-Methylnaphthalene	90-12-0	142	141	
8.) * Acenaphthene-d10	15067-26-2	164	162	160
9.) $ 2-Fluorobiphenyl	321-60-8	172	171	
10.) Acenaphthylene	208-96-8	152	151	153
11.) Acenaphthene	83-29-9	154	153	152
12.) Fluorene	86-73-7	166	165	167
13.) * Phenanthrene-d10	1517-22-2	188	94	80
14.) Phenanthrene	85-01-8	178	179	176
15.) Anthracene	120-12-7	178	179	176
16.) Fluoranthene	206-44-0	202	101	203
17.) Pyrene	129-00-0	202	200	203
18.) * Chrysene-d12	1719-03-5	240	120	236
19.) $ Terphenyl-d14	1718-51-0	244	122	212
20.) Benzo(a)anthracene	56-55-3	228	229	226
21.) Chrysene	218-01-9	228	226	229
22.) * Perylene-d12	1520-96-3	264	260	265
23.) Benzo(b)fluoranthene	205-99-2	252	253	125
24.) Benzo(k)fluoranthene	207-08-9	252	253	125
25.) Benzo(a)pyrene	50-32-8	252	253	125
26.) Indeno(1,2,3-cd)pyrene	193-39-5	276	138	277
27.) Dibenz(a,h)anthracene	53-70-3	278	139	279
28.) Benzo(g,h,i)perylene	191-24-2	276	138	277

* Internal standard compound
$ Surrogate compound

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APPENDIX _8: DFTPP TUNING

DFTPP Key Ions and Ion Abundance Criteria
8270C Semivolatile Organics by GC/MS

Mass	Ion Abundance Criteria
51	30.0% to 60.0 % of mass 198
68	< 2.0% of mass 69
69	(reference only)
70	< 2.0% of mass 69
127	40.0% to 60.0% of mass 198
197	< 2.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0% to 9.0% of mass 198
275	10.0% to 30.0% of mass 198
365	> 1.0% of mass 198
441	Present but less that mass 443
442	> 40.0% of mass 198
443	17.0% to 23.0% of mass 442

Tune acceptance is based on an average of three consecutive scans at the apex with background subtraction. If the DFTPP does not pass using these options, another DFTPP should be analyzed. If that also fails, instrument maintenance should be performed to correct the problem. *No sample data associated with a failing tune standard may be reported.*

This document contains information that may only be disseminated to C&T staff, clients & regulators.

APPENDIX_9: **MINIMUM RESPONSE CRITERIA**
For EPA 8270D

Compound	Minimum Response Factor
1,4-Dioxane	0.05
Naphthalene	0.7
1-Methylnaphthalene	0.4
2-Methylnaphthalene	0.4
Acenaphthylene	0.9
Acenapthene	0.9
Fluorene	0.9
Phenanthrene	0.7
Anthracene	0.7
Fluoranthene	0.6
Pyrene	0.6
Benzo(a)anthracene	0.8
Chrysene	0.7
Benzo(b)fluoranthene	0.7
Benzo(k)fluoranthene	0.7
Benzo(a)pyrene	0.7
Indeno (1,2,3,-cd)pyrene	0.5
Dibenz(a,h)anthracene	0.4
Benzo(g,h,i)perylene	0.5

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Curtis & Tompkins, Ltd.

SEMIVOLATILE ORGANIC COMPOUNDS

(Base/Neutrals & Acids by GC/MS)
EPA 8270C, EPA 8270D & EPA 625

Approved by:	Signature:	Date:
Cheryl Pacheco Operations Manager	for CP	6/7/13
Teresa Morrison QA Director		6/7/13
Reapproved:		

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Curtis & Tompkins, Ltd.

SEMIVOLATILE ORGANIC COMPOUNDS

(Base, Neutral & Acid Extractable Organics by GC/MS)
EPA 8270C, EPA 8270D & EPA 625

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SEMIVOLATILE ORGANIC COMPOUNDS
(Base, Neutral & Acid Extractable Organics by GC/MS)
EPA 8270C, EPA 8270D & EPA 625

1.0 SCOPE

This procedure describes the GC/MS identification and quantitation of basic, neutral, and acidic organic compounds that have been previously extracted from soil, water, and miscellaneous matrices. These compounds may be quantified down to 10-50 ug/L in liquid samples and 67-1,700 ug/Kg in soil samples (see Appendix 10 for reporting limits of specific compounds).

EPA 8270 was written by the EPA's Office of Solid Waste with additional guidance for surface water and ground water, as opposed to EPA 625 was written by the EPA's Office of Water specifically for wastewater. EPA 625 may also be requested for groundwater samples if the client is planning to discharge the water, with or without additional treatment, into a wastewater stream or into naturally occurring surface waters (bay or river). See Appendix 13 for EPA 625 requirements.

2.0 REFERENCES

Analytical Methods:
EPA 8000B, *Determinative Chromatographic Separations*, SW-846, Dec 1996
EPA 8000C, *Determinative Chromatographic Separations*, SW-846, March 2003
EPA 8270C, *SVOCs by GC/MS*, SW-846 Update 3, December 1996
EPA 8270D, *SVOCs by Gas Chromatography/Mass Spectrometry,* SW846, Feb 2007
EPA 625, Base/Neutral and Acids, 40CFR136 Appendix A

Extraction Methods:
EPA 3500B, Organic Extraction & Sample Preparation, SW-846, Dec 1996
EPA 3500C, Organic Extraction & Sample Preparation, SW-846, Feb 2007
EPA 3510C, Separatory Funnel Liquid-Liquid Extraction, SW-846, Dec 1996
EPA 3520C, Continuous Liquid-Liquid Extraction, SW-846, Dec 1996
EPA 3540C, Soxhlet Extraction, SW-846, Dec 1996
EPA 3550C, Ultrasonic Extraction, SW-846, Feb 2007
EPA 3580A, Waste Dilution, SW-846, July 1992

Other References:
C&T SOP QA 1.5, *Calibrating & Maintaining Temperature Controls*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL)*
C&T SOP QA 4.6, *Limit of Quantitation (LOQ)*
C&T SOP QA 9.6, *Insuring Compliant Manual Integration*
Operator Manuals for HP5890, Agilent 6890, and Agilent 6892 GC's
Operator Manuals for HP5971, HP5972, HP5973, and Agilent 5975 MSD's
Manuals for "Chemstation" software and operating system
Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Version 4.2, Oct 2010

This SOP contains information that may only be disseminated to C&T staff, clients & regulators.

DoD Quality Systems Manual, Version 5.0 Final Draft, Mar 2013

3.0 PRESERVATION & HOLDING TIMES

Samples: No chemically preservation.
Store at > 0.0 to 6.0°C
Water samples should be collected in amber glassware and stored protected from light

Water: Extraction must begin within 7 days of collection.
Soil: Extraction must begin within 14 days of collection.

Extracts: Store methylene chloride extracts in a freezer at < -10° C.
Analyze within 40 days of extraction.

4.0 SAFETY

Assume all standards, samples, and extracts may contain hazardous and/ or potentially toxic chemicals.

5.0 QC REQUIREMENTS

The extraction lab will prepare a Method Blank (MB), Laboratory Control Sample (LCS), Matrix Spike and Matrix Spike Duplicate (MS/MSD) with each batch of twenty or fewer samples. If there is insufficient sample volume to prepare an MS/MSD, a Blank Spike (BS) and Blank Spike Duplicate (BSD) will be prepared in place of the LCS/ MS/ MSD. Every extract will contain surrogate compounds, which are added prior to extraction to monitor the performance of the extraction. The analyst must add an internal standard mix to each extract before analysis, to monitor the performance of the instrument. See the associated SOP *8270C Laboratory Control Limits Table-1* for current in-house surrogate and spike recovery limits.

A DFTPP tune standard must be analyzed and pass acceptance requirements at the beginning of each 12-hour tune-shift, before any other standards or extracts are analyzed. A Continuing Calibration Verification (CCV) standard must be analyzed immediately after a passing tune standard and must pass acceptance criteria for all analytes applicable to the samples to be subsequently analyzed within the 12-hour tune-shift. See Appendix 6 for DFTPP acceptance criteria and Appendix 7 for CCV acceptance criteria.

PEM: A Performance Evaluation Mix (PEM) standard must be analyzed with each 12-hour tune shift that contains samples from any project associated with the Department of Defense (DoD) Quality Systems Manual (QSM) or samples submitted for EPA 625.

An initial calibration curve must be established for every analyte from a minimum of 5 points, using internal standard calibration. The concentration of the lowest point must be at or below the reporting limit and that of the highest point determines the upper limit of quantitation. A mid-range Initial Calibration Verification (ICV) standard obtained from a

second manufacturer must be analyzed immediately following the ICAL standards, before any samples are analyzed; the ICV must meet CCV acceptance limits. Sample results associated with a failing initial calibration cannot be reported. See Appendix 8 for acceptance criteria.

A Method Detection Limit (MDL) study will be extracted and analyzed whenever new instruments are installed or significant changes are made to the procedure. Limit of Detection (LOD) and Limit of Quantitation (LOQ) samples are extracted and analyzed quarterly. In-house QC limits are updated every 6 months based on control-charts of data from the previous year. See associated QA SOPs for details.

Note: Samples may be associated with a client-prepared Quality Assurance Project Plan (QAPP) that contains different requirements than those listed in this SOP. For those samples, the QAPP requirements supersede those listed in this document.

6.0 INTERFERENCES

6.1 Some of the target compounds for this analysis are relatively volatile and may be lost if the extract is superheated or allowed to go dry during extraction.

6.2 Bis(2-ethylhexyl)phthalate is a common laboratory contaminant that can easily be introduced during the extraction process if gloves or other plastics come into contact with the solvent.

6.3 High levels of organic acids will destroy the column stationary-phase; organic acid peaks can be recognized by their characteristic 'fronting'. If the phenols responses drop drastically after analysis of a sample with high concentrations of organic acids, the column will probably need to be replaced.

6.4 Heavy oils and tars, which give extracts a deep coffee color, will cause active sites in the injection port and analytical column; poor internal standard recoveries (primarily perylene-d12 but also chrysene-d12 to a lesser extent) will occur in viscous, oily extracts. High levels of other non-target analytes may mask the presence of the target compounds. See the 'Dilutions' section below for further discussion of these interferences and appropriate analytical procedures.

6.5 If Aniline, Benzidine and/or Pyridine is on the target analyte list for liquid samples, the secondary, basic (pH>11 SU) extraction must be performed. These samples should be logged in for "8270-2" or "625-2", where the "-2" indicates that both phases are needed.

6.6 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

6.7 Pyridine may perform poorly at the GC injection port temperatures listed in this method. Lowering the injection port temperature may reduce the amount of degradation. However, the analyst must use caution in modifying the injection port temperature, as the performance of other analytes may be adversely affected. Therefore, if pyridine is to be

determined in addition to other target analytes, it may be necessary to perform separate analyses. In addition, pyridine may be lost during the evaporative concentration of the sample extract. As a result, many of the extraction methods listed for this procedure may yield low recoveries unless great care is exercised during the concentration steps.

6.8 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

6.9 N-Nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions commonly used for this method.

6.10 N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. For this reason, it is acceptable to report the combined result for N-Nitrosodiphenylamine and Diphenylamine for either of these compounds as a combined concentration.

6.11 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to Azobenzene. Given the stability problems, it would be acceptable to calibrate for 1,2-diphenylhydrazine using Azobenzene. Under these poor compound separation circumstances the results for either of these compounds should be reported as a combined concentration.

6.12 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

6.8 If Pesticides are included in the target analyte list: Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, γ-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected to be present

7.0 EQUIPMENT (Refer to Appendix 4 for specific instrument configurations)

Autosamplers:

HP 7673 ALS, HP 7683 ALS

Gas Chromatographs:

Models HP5890, HP6890, Agilent 6890
Fused Silica Capillary Column 30m x 0.25mmID x 0.25 – 0.50μm df
Electronic Pressure Control

Mass Spectrometers:

Models HP5971, HP5972, HP5973, HP5975

Software & Hardware:

Dell Dimension XPS P90 DOS PC with Chemstation Software
Windows 95 & NT servers with Chemstation Software
Windows 95 & NT servers with Target software

8.0 DAILY MAINTENANCE

The septum, injection liner, guard column, and blue seal should be changed after every sequence of sample extracts run and before the analysis of the DFTPP/PEM solution. If this daily maintenance is **not** performed, make a notation on the sequence summary in LIMS to that effect. The injection liner is a 4mm splitless liner packed with a small amount of silanized glass wool. The analytical column will need to be trimmed (usually a minimum of 15cm) if oily or highly contaminated samples were analyzed in the previous tune-shift; additional trimming of the column may be necessary, depending on the results of the analysis of the DFTPP tuning mix standard and a Continuing Calibration Verification (CCV) standard. If the analytical column is trimmed, document it in the maintenance log. See Appendix 5 for additional maintenance procedures & details.

9.0 DAILY INSTRUMENT SEQUENCE

Each sequence must begin with a DFTPP Tuning standard followed by a Continuing Calibration Verification (CCV) standard. Once the tune, CCV, and PEM (if required) have passed acceptance criteria, sample extracts may be added to the instrument sequence. An example of a typical instrument sequence is presented below. *All samples (and associated batch QC) must be injected within 12 hours of the associated DFTPP injection time*. Approximately 15-24 runs can be completed within the 12 hour period. An example of a typical sequence is presented here.

1. DFTPP/PEM 50 ng
2. CCV Standard
3. Method Blank
4. LCS or BS/BSD (if applicable)
5. MSS Sample
6. Matrix spike (MS)
7. Matrix spike duplicate (MSD)
8. Samples

9.1 Writing a Chemstation Sequence

Various user reports are automatically produced after the run is complete. This automation is based on the type of sample being analyzed, the sample number, the LIMS identification of any associated calibration or spiking standards, the batch number, and any applicable dilution factors. This data must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information and should be written into the sequence as follows:

TUN, S#
CCV, S#
MB, QC#, Batch#, IDF
LCS, QC#, Batch#, IDF
S, Samplenum, Batch# , IDF
MS, QC#, Batch#, IDF
MSD, QC#, Batch#, IDF

Where:
S# is the LIMS WS# of the standard used,
QC# is the LIMS ID of the batch QC sample (ie: QC169359)
IDF is the instrument dilution factor for the sample, written as “1000x” or similar
Samplenum is the LIMS sample number (ie: 160961-005)

Additional comments regarding the extracts or standards can be added by placing two spaces followed by two forward slashes (“//”) and the comments that should appear on the user reports. LIMS recognizes shorthand for some of the most common comments such as:

DV (dark and viscous)
Viscous
NT (high non-targets)
Amber
Dark

9.2 Data File Naming Conventions

Data file names are based on a maximum field length of 6 characters and so must use abbreviated codes for the instrument ID and date. The instrument ID, month, and day codes are outlined below. Data files are named as:

IMD## where I = Instrument ID, M = Month, D = Day, ## = Run Number (01 through 99).

Instrument	Designator	Instrument	Designator
BNA02	U	BNA06	Y
BNA03	V	BNA07	Z
BNA04	W	BNA09	R
BNA05	X		

Month	Designator	Month	Designator	Month	Designator
January	A	May	E	September	I
February	B	June	F	October	J
March	C	July	G	November	K
April	D	August	H	December	L

Day	Designator	Day	Designator	Day	Designator
1	1	11	B	21	L
2	2	12	C	22	M
3	3	13	D	23	N
4	4	14	E	24	O
5	5	15	F	25	P
6	6	16	G	26	Q
7	7	17	H	27	R

8	8	18	I	28	S
9	9	19	J	29	T
10	A	20	K	30	U
				31	V

For example, the data file for the 2nd run on instrument BNA03 on October 28 would be named VJS02 (instrument V, October, 28th, run 02).

9.3 Tuning (See Appendix_6 for DFTPP Acceptance Criteria):
The MS-detector must be tuned to meet specific performance criteria so that data produced by this instrument will be comparable to that produced by another. Analyze a 50 µg/mL Decafluorotriphenylphosphine (DFTPP) standard at the beginning of each 12-hour tune-shift, and meet the acceptance criteria listed in Appendix_6, before any other standards or extracts are analyzed. Samples injected outside the 12-hour clock cannot be reported.

9.3.1 Start the daily sequence (or 2nd 12-hour tune shift) by analyzing the DFTPP, prior to analysis of the calibration verification standards and samples. Use the method DFTPP##.m, where ## is the instrument number (ie: 02).

9.3.2 See Appendix_2 for instructions on preparing the 50ng aliquot of DFTPP (Decafluoro triphenyl phosphine).

9.3.3 After the DFTPP has run, use the software “autofind” process to generate a report comparing the mass spectrum to the performance criteria specified in EPA 8270C (see Appendix 6) using the average of the three scans (apex plus one on either side) and background subtraction. Alternately, you can specify a single scan, the average of two consecutive scans or the average of three consecutive scans. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak. If the DFTPP fails acceptance criteria, the failing range will be flagged with an asterisk. If no asterisk appears on the report, the DFTPP meets tune criteria; continue by preparing and analyzing a CCV.

9.3.4 If the DFTPP fails criteria (listed in Appendix_6), verify conditions and set-points, and reanalyze the DFTPP. If the second shot fails, tune the instrument, and/or schedule preventative maintenance including source cleaning. *Major* maintenance (excluding daily maintenance such as clipping the column, changing the liner, etc.) should be recorded in the instrument maintenance log. See Appendix_5 for trouble-shooting and preventative maintenance guidance. *Sample results associated with the failing 12-hour tune-shift cannot be reported.*

9.4 CCV (Continuing Calibration Verification) (See Appendix 7 for acceptance criteria): After the DFTPP passes tune criteria, analyze a CCV (Continuing Calibration Verification) standard to verify that the response of the instrument has not changed significantly and that the curve may still be used to quantitate sample results. This standard must be run at the beginning of each 12-hour tune-shift, immediately after the DFTPP, using a standard at one of the middle levels of the calibration curve; do not use either the highest or lowest point. The standard concentration used for the CCV should be alternated over the course of several tune shifts.

9.4.1 Decide what concentration CCV standard to analyze; concentrations should be alternated across the mid-levels of the calibration curve.

9.4.2 See Appendix 2 for instructions on preparing the CCV standard.

9.4.3 Analyze this standard using the same data acquisition method as for the samples, typing “CCV,” before the working standard number.

9.4.4 After the analysis is complete, examine the integration and spectra to ensure that the correct peaks were picked and integration is correct for each compound.

9.4.5 Send the data to LIMS to generate and print a CCV summary, comparing the response factors from this standard to those of the ICAL curve.

9.4.6 Examine the CCV summary against the following criteria to determine whether the CCV is acceptable:

a. Per method 8270D: Evaluate the retentions times of the internal standards in the continuing calibration verification standard for any retention time shifts by more than 30 seconds from the internal standard in the midpoint of the most recent ICAL. If a retention time shifts by more than 30 seconds, inspect the chromatographic system for malfunction; after corrections are made to the system, reanalyze samples analyzed while the system was malfunctioning. Routine maintenance, such as clipping the column, is not considered a chromatographic system malfunction. When retention times shift by more than 30 seconds due to routine maintenance, document it in the maintenance bench book and narrate it in LIMs on the CCV page with the bench book number and page.

b. Per method 8270D: Evaluate the area of the internal standards against the midpoint of the most recent calibration. The area of each internal standard must fall between 50-200% of the ICV.

c. The retention times should increase with assigned ‘compound number’ (in the acquisition software). The compound spectra should be re-examined for any RT’s that appear out of order.

d. All compounds must meet minimum relative response factor (RRF) of 0.05.

8270D: For sequences containing samples run for 8270D, the CCV minimum RRF must meet those listed in Appendix 14, which are taken from the updated method.

e. **SPCC** (System Performance Check Compounds) RRF must be ≥ 0.05.

n-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocylclopentadiene	4-Nitrophenol

8270D: Method 8270D does not discuss SPCCs but instead requires each target analyte to be evaluated. For sequences containing samples run for 8270D, the CCV minimum RRF must meet those listed in Appendix 14, which are taken from the updated method.

f. **CCC** (Calibration Check Compounds) percent difference (or percent drift if the compound was calibrated using linear regression) from the initial calibration must be ≤ 20%.

Acenaphthene	4-Chloro-3-methylphenol
Benzo(a)pyrene	2,4-Dichlorophenol
1,4-Dichlorobenzene	2-Nitrophenol
Fluoranthene	Phenol
Hexachlorobutadiene	Pentachlorophenol
N-Nitrosodiphenylamine	2,4,6-Trichlorophenol
Di-n-octyl phthalate	

8270D: Method 8270D does not include discussion of CCC compounds but requires each target analyte to be evaluated; see the Non-CCC discussion below.

g. **Non-CCC** compounds must have %D ≤ 30%, with the exception of poor performers (such as Hexachlorocyclopentadiene, the Nitroanilines, Benzidine, etc.) that must have %D ≤ 40%.

625: For sequences containing samples run for EPA 625, the CCV %D for each compound should be ≤ 20% as this method does not discuss minimum response requirements or differentiate between various types of analytes.

8270D: For sequences containing samples run for 8270D, the CCV %D should be ≤ 20%.

h. 8270D: The updated method requires that isomeric pairs have sufficient resolution between the peaks in order to report each compound individually.

The height of the valley between the two peaks must be less than 50% the average height of the two peaks. If this resolution is *not* demonstrated in the CCV the two isomers must be integrated together and reported as one compound for all runs in the sequence.

9.4.7 If any of the above criteria are not met, re-examine the integration to verify that each peak was correctly integrated. Manual integrations must be consistently applied to ICAL, CCV, batch QC, and sample integrations. If manual integrations are performed, resend the file to LIMS so that a corrected CCV summary can be generated.

Unsubstantiated alteration of peak integration solely to pass calibration or QC criteria is illegal and is grounds for immediate termination.

9.4.8 If the SPCC and CCC criteria are not met, another CCV standard should be analyzed. If the second analysis of the standard fails to meet the SPCC and CCC criteria, recalibration and/or other instrument maintenance is required.

If two CCV's were analyzed, LIMS will use the most recent file. *Do not* "cherry pick" some compounds from the first CCV and others from the second CCV; if the second CCV is processed and used, all compounds must be taken from the second standard.

9.4.9 If the SPCC and CCC criteria are met, but other compounds fail acceptance criteria, data may be reportable based on the following criteria:

a.) If the failing compound is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b.) If RRF fails the minimum RRF requirement (RRF < 0.05) and is a required target compound for the associated samples, the samples must be reanalyzed.

c.) If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

d.) If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.

e.) If the compound fails the %D criterion due to a low response and *was* detected (even below the reporting limit), the sample must be reanalyzed.

8270D: The updated method does not differentiate between CCC and non-CCC compounds, all compounds of concern must have ≤ 20%D, and no more than 20% of all calibrated compounds may fail in the CCV. Individual compounds may fail low, but "in order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable quantitation limit".

See Appendix_1 for calculation of %D and the relative response factor (RRF).

9.5 PEM (Performance Evaluation Mix)

For any EPA 625 samples or 8270 samples associated with the DoD QSM, analyze a 50 µg/mL PEM (Performance Evaluation Mix) standard containing Benzidine, 4,4'-DDT, and Pentachlorophenol. This standard is used to evaluate 1.) injector port inertness by verifying that the DDT breakdown to DDE and DDD is within acceptance criteria and 2.) column performance by demonstrating that benzidine and pentachlorophenol do not exhibit pronounced tailing.

625: EPA 625 does not require inclusion of DDT in the PEM standard.

Breakdown: DDE and DDD (DDT breakdown products) should not be present, either individually or combined, at levels greater than 20%. If this criterion is not met, maintenance should be performed on the injection port. See Appendix_1 for the breakdown calculation.

Tailing: EPA 8270C requires a visual confirmation of acceptable peak shape however method EPA 625, 8270D, and DoD QSM require mathematical verification. For PEM's associated with DoD samples, the tailing factor should be < 2 for Pentachlorophenol and Benzidine. If these criteria are not met, additional maintenance (column clipping) should be performed. See Appendix_1 for the tailing calculation.

9.6 Prepare the Sample and Batch QC Extracts for analysis

9.6.1 Let the extracts warm to room temperature then aliquot 100uL of extract into an autosampler vial.

Note: If the extract is black & oily or opaque, make a dilution that will result in an extract that is very dark amber in color. If black, oily, or viscous extracts are analyzed, a tarry residue will build up in the injection port, causing active sites and low recoveries for the Perylene-d12 internal standard.

9.6.2 Add 1uL of the internal standard solution and seal the vial with a crimp cap.

9.6.3 If a 2x dilution is required, dilute a 100 µL aliquot of extract with 100 µL methylene chloride, then spike with 2 µL of internal standard solution, and cap.

9.6.4 If the required dilution is >3x, dilute an aliquot of the extract to a total volume of 500uL, add 5uL internal standard solution, and cap.

9.6.5 Place the samples on the autosampler tray beginning with the lightest colored extracts followed by more highly colored or viscous extracts.

10.0 QUANTITATIVE ANALYSIS

10.1 Sample Quantitation

Analyte quantitation is done using the internal standard technique. For semivolatile analysis by GC/MS, six internal standards are used. Quantitation is based on comparison of the area of the primary ion in the sample's mass spectra to the initial calibration response factor for that compound. An Internal Standard (ISTD) technique is used to correct for slight autosampler injection variances and some types of matrix interferences. See Appendix_1 for example calculations.

A user report will be automatically generated once the run is complete. Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

Creosote: Clients may occasionally request 'creosote'. For these jobs, analyze a Coal Tar standard and identify 5 primary PAH peaks. These five peaks must be present in the sample to report a detected quantity of creosote. Quantitation is then based on the concentration of Phenanthrene as determined against the creosote standard.

10.2 Dilutions

10.2.1 If the extract is black, oily & viscous, or opaque, make a dilution that will result in an extract that is very dark amber in color. If black, oily, or viscous extracts are analyzed, a tarry residue will build up in the injection port, causing active sites and low recoveries for the Perylene-d12 internal standard.

10.2.2 If a single target compound(s) is within 10x the upper calibration limit of the instrument, prepare a dilution that will bring the over-range compound into the upper half of the calibration range. Report the majority of the target compounds from the first analysis and the over-range compounds from the in-range dilution.

10.2.3 If the extracts are not dark & viscous, the dilutions should be made so that the highest target compound falls within the upper-half of the calibration curve. See Appendix_3 for preparing various dilutions.

10.2.4 If the sample chromatogram exhibits a typical fuel "hump", analyze the sample at a dilution that will bring the "hump" baseline to approximately half the height of the internal standard peaks. Do not try to analyze it at much higher levels, as the background hydrocarbons may obscure target compounds. On the Data Review Checklist, narrate raised reporting limits and, if low recovery for the Perylene-d12 was observed, internal standard recovery failure as due to obvious chromatographic interferences.

10.2.5 If the sample chromatogram includes a large but narrow non-target peak, analyze the sample at a dilution that will bring the non-target peak to no more than 5 times the height of the internal standard peaks. On the Data Review Checklist, narrate raised reporting limits as due to non-target matrix interferences.

10.2.6 If the sample chromatogram includes a very wide non-target peak, be aware that this peak may obscure target compounds or shift retention times of later-eluting compounds and dilute accordingly; discuss the problem with the Department Manager or QA Director. On the Data Review Checklist, narrate raised reporting limits as due to non-target matrix interferences.

10.2.7 If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

10.3 Surrogates

Surrogate compounds are chemically similar to the target analytes but are compounds not found in actual samples. These compounds are added, prior to extraction, to every sample, method blank, and spike to monitor the efficiency of the extraction for that sample. In-house Surrogate Acceptance Criteria are specified in the associated SOP '*8270C Laboratory Control Limits, Table-1*'. These limits are generated annually, using control charts.

Acid Surrogate Compounds:	Base/ Neutral Surrogate Compounds:
2-Fluorophenol	Nitrobenzene-d5
Phenol-d6	2-Fluorobiphenyl
2,4,6-Tribromophenol	Terphenyl-d14

10.3.1 After each sample is analyzed, LIMS will automatically generate a user report with the surrogate criteria for that sample and flag any failing recoveries. If a surrogate recovery is outside QC limits, determine whether reanalysis is required using the following criteria:

a. If all of the surrogate recoveries fail acceptance limits, verify that the LIMS S#, amount, and concentration of surrogate added to the sample is correct. If these are correct, the sample must be reanalyzed. If the surrogates again fail, the samples must be re-extracted.

b. If a high recovery is observed but no target analytes were detected above the reporting limit in the sample, note the failure on the 'Data Review Checklist' and report the data without reanalysis, since the possible high bias will not affect sample results.

c. If a high recovery is observed and target analytes were detected, and there is no obvious chromatographic interference, the sample must be reanalyzed. If the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. If the same surrogate fails criteria after re-extraction it is deemed to be matrix effect. Include both sets of data in the package and note the situation in the case narrative.

d. If a low recovery was observed but the client requested only a subset (acid = 8040 or base/neutral = 8100) of analytes and the failing surrogate is not associated with that fraction, the data may be reported without reanalysis.

e. If a low recovery is observed for the first and/or second acid surrogates and the sample chromatogram indicates the presence of high levels of organic acids, the data may be reported without reanalysis or re-extraction, so long as the samples are not associated with a QAPP requiring re-extraction. Document the surrogate failure on the 'Data Review Checklist' and narrate the failure as due to matrix interferences ("organic acids").

f. If a low recovery is observed for any surrogate and there is no obvious chromatographic interference, or documented historical site matrix interference, the sample must be reanalyzed. If the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. If the same surrogate fails criteria after re-extraction it is deemed to be matrix effect. Include both sets of data in the package and note the situation in the case narrative.

10.3.2 If a sample must be re-extracted and the holding time has expired, the client's Project Manager must log the sample into LIMS as an alias and have the sample re-extracted as the new sample number. If the sample is still within holding time, the sample should be re-extracted under the original sample number.

10.3.4 If upon re-extraction, the surrogate recovery is again outside limits, note the matrix effect as "confirmed matrix interference" on the User Report and the Data Review Checklist, otherwise report the data with passing surrogate recovery.

Note: Project-specific quality assurance project plans (QAPPs) may require batch control based on different compounds and control limits, in which case the project requirements supersede this SOP for all samples related to that project.

10.4 Internal Standards

Internal standard compounds are chemically similar to the target analytes but are compounds not found in actual samples. These compounds are added by the analyst to each sample, batch QC sample, and standard to correct for slight autosampler injection variances and some types of matrix interferences. Quantitation is based on comparison of the area of the primary ion in the sample's mass spectra to the initial calibration response factor for that compound. See Appendix_1 for example calculations and Appendix_9 for target compound/ internal standard assignments.

The following internal standards are added to all sample and QC extracts at 40ug/mL after extraction and prior to analysis:

1,4-Dichlorobenzene-d4	Naphthalene-d8
Acenaphthene-d10	Phenanthrene-d10
Chrysene-d12	Perylene-d12

10.4.1 Review the internal standards results against the following criteria:

a. The retention time of the internal standards must be ± 0.5 minutes from the CCV.

b. The area of each internal standard must fall between 50-200% of the mid-point of the ICAL.

10.4.2 If internal standard recoveries are out of compliance, use the following to determine the appropriate corrective action:

a. If the internal standard is failing in a calibration standard (ICAL, ICV, CCV), instrument maintenance must be performed to bring the instrument back into compliance. The standard and any extracts run after that standard must be rerun.

b. If, upon visual inspection of chromatograms, matrix interference is apparent (ie: background is 2 to 5 times higher than the nearest internal standard) and no target analytes were detected, the data may be reported without corrective action. Narrate the interference.

c. If obvious matrix interference is not present, the sample must be reanalyzed.

d. If, upon reanalysis, the same internal standard falls outside QC limits, report the better of the two runs and narrate the failure as due to confirmed matrix interference.

8270C Method Modification Note: Internal standard retention time criteria are based on the daily calibration (CCV), not the mid-point of the initial calibration as noted in 8270C. This approach follows the guidance listed in 8000B and earlier versions of 8270, as the column

is trimmed daily and retention times routinely differ from those observed in the initial calibration.

10.5 Batch QC

For every batch of 20 samples (or less) analyzed, a Method Blank (MB), a Laboratory Control Sample (LCS), a matrix spike (MS) and duplicate (MSD) are extracted and analyzed. If insufficient sample volume was submitted for matrix QC, a blank spike (BS) and blank spike duplicate (BSD) are extracted in place of the LCS/ MS/ MSD.

Note: Project-specific quality assurance project plans (QAPP's) may contain different requirements than those listed in this SOP. If so, the QAPP requirements supersede this SOP for all samples related to that project.

10.5.1 **Method Blank (MB):**

A method blank is extracted with each batch of samples to verify that the extraction reagents and process are not contributing to the sample results. No compounds should be detected in the method blank, however if a compound(s) is detected, the following steps are used to determine the corrective action required:

a. If the compound(s) is a common lab contaminant (ie: bis(2-ethylhexyl)phthalate) and the result is less than the reporting limit, document the contamination on the batch sequence summary, the data package narrative, and the review checklist and report the data without reanalysis.

b. If the compound(s) is not a common lab contaminant and the concentration is between the reporting limit and 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

c. If the sample result for that compound(s) is greater than ten (10) times the amount found in the method blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

d. If the sample result for that compound(s) is greater than the reporting limit but less than ten (10) times the amount found in the associated method blank, the samples must be re-extracted and reanalyzed.

10.5.2 **Laboratory Control Sample (LCS, or BS/BSD pair):**

Laboratory Control Samples are extracted with each batch of samples to demonstrate the performance of the extraction and analysis in the absence of matrix interferences. The spiked concentrations should fall in the low to mid-range of the calibration curve; if normal initial calibration ranges are changed, review the spiking levels against the new ranges to evaluate whether the spiking levels need to be changed as well. In-house Acceptance Criteria are specified in the associated

SOP '*8270C Laboratory Control Limits, Table-1*'. These limits are generated annually, using control charts.

SW-846 Method 3500, Section 5.5.1 defines required spike compounds as the following:

Phenol	Pentachlorophenol	Acenaphthene
2-Chlorophenol	1,4-Dichlorobenzene	2,4-Dinitrotoluene
4-Chloro-3-methylphenol	N-Nitroso-di-n-propylamine	Pyrene
4-Nitrophenol	1,2,4-Trichlorobenzene	

Sporadic marginal failures are allowed for 5% (4 compounds) of the target compound list, except that the compounds listed in EPA 3500B Section 5.5.1 must pass acceptance criteria. The sporadic failures must be random failures; if the same analyte fails in 3 consecutive batches, start a Corrective Action Report then investigate and correct the source of the problem.

10.5.2.1 After all of the samples in the batch have been run, the "reduced" LCS report can be generated to determine if the LCS passed acceptance criteria for all of the client-specified limits associated with the batch; this report will compare the recoveries (and RPD for BS/BSDs) for each compound to the tightest limits applicable to the jobs in that batch. To run this report:

Open the LIMS Intranet page and go to the GC/MS BNA Instrument Page.

There are a number of buttons In the middle of the page. Enter the batch number in the field next to "View QC status for batch" then click the VIEW button.

10.5.2.2 If any of the 11 compounds listed above (EPA 3500 compounds) or more than 3 of the remaining compounds fail acceptance criteria, reanalyze the QC extracts. If the failure is confirmed upon reanalysis, initiate a Corrective Action Record and use the following criteria to determine the required corrective action:

a. If the samples are being analyzed for only a subset of the target compound list (8100, 8040, etc) and all of those compounds pass acceptance criteria, the data may be reported without further corrective action.

b. If high recoveries are observed but no target analytes were detected in the associated samples, note the failure on the Data Review Checklist and report the data without re-extraction, as the potential high bias does not affect the sample results.

c. If high recoveries are observed and the samples contain target compounds at levels above the reporting limits, the samples must be re-extracted.

d. If high RPD's are observed but the recoveries are within acceptance limits and no target analytes were detected in the associated samples, note the failure on the Data Review Checklist and report the data without re-extraction, as the lack of good precision data does not affect ND samples.

e. If high RPD's are observed and the samples contain target compounds at levels above the reporting limits, the samples must be re-extracted.

f. If low recoveries are observed for any surrogate, the associated samples must be re-extracted.

10.5.2.3 If a sample must be re-extracted and the holding time has expired, the client's Project Manager should log the sample in as an alias and have the samples re-extracted as the new sample number. If the sample is still within holding time, re-extract and reanalyze the sample under the original sample number.

10.5.3 **Matrix Spike/ Matrix Spike Duplicate (MS/MSD):**
Matrix spikes are extracted with each batch of samples to demonstrate the accuracy (recovery) and precision (RPD) of the analysis in real-world samples. In-house Acceptance Criteria are specified in the associated SOP '*8270C Laboratory Control Limits, Table-1*'. These limits are generated annually, using control charts. The matrix spikes include all analytes of interest, but are typically monitored only for those compounds listed in method 3500B Section 5.5.1, unless otherwise specified by the client.

10.5.3.1 Review the MS/MSD data. If either the recoveries or RPD fail criteria, determine whether or not the data can be reported based on the following:

a. If the concentration of a target compound in the sample is greater than the linear range and the sample needs to be rerun for just that compound, report the MS/MSD with a LIMS-flag of ">LR" on those recoveries without reanalysis.

b. If the concentration of a target compound in the sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a ">LR" flag to those recoveries. Report the data without reanalysis.

c. If the concentration of a target compound is greater than 4x the spiking level, LIMS will apply a “NM” (for “Not Meaningful”) flag to those recoveries. Report the data without reanalysis.

d. If MS/MSD recoveries fail but the RPD (and LCS recovery) is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

e. If the recoveries fail due to obvious chromatographic interference (ie: coelution of sample hydrocarbons or other analytes with the spike compounds), narrate the failure on the Data Review Checklist and report the data without reanalysis.

f. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

11.0 QUALITATIVE ANALYSIS (Target Compound Identification)

Identification of compounds is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference spectrum.

The reference spectrum for target compounds is generated from the initial calibration standards. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

The following criteria need to be met for positive identification of a compound:

11.1 The retention time (RT) must be within 0.1 minute of that component in the most recent CCV. If co-elution of interfering components prohibits accurate assignment of sample RRT, the RRT should be assigned from extracted ion current profiles of ions unique to the compound of interest.

Method Modification Note: Method 8270C Section 7.6.1.2 specifies that the retention time be within 0.06 RRT, however this window is too narrow for routine use with highly contaminated samples. C&T has chosen to use a default of 0.1 minutes.

Method Modification Note: Due to the daily maintenance requirements of this method, the retention times are based on the most recent CCV (8000B Section 7.6.5, 8000C Sect.11.6.5, QSM5.0-FD Table 4, and previous versions of method 8270), *not* on the mid-point of the initial calibration (8270C, Section 7.4.6).

11.2 The retention time of the associated internal standard must be within ± 0.5 minutes of the internal standard retention times in the most recent CCV.

11.3 The retention times of the characteristic ions of the compound should agree.

11.4 All ions of greater than 10% intensity, relative to the quantitation ion, must be present in the sample spectrum.

11.5 The relative intensities of the characteristic ions (for target analytes and tentatively identified compounds, “TICs”) agree within 20% of the same ions in the reference spectrum.

Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance must be between 30-70%.

If a client requests “TICs” (Tentatively Identified Compounds), see Appendix 11 for instructions on processing and reviewing the data.

11.6 Structural isomers may be reported as individual isomers so long as the height of the valley between the two peaks should be less than 25% of the sum of the two peak heights, otherwise they should be identified as isomeric pairs (8270C, Section 7.6.1.4).

In most samples, if Benzo(b)fluoranthene is present, there will also be a peak for Benzo(k)fluoranthene. If only a single peak is present and the retention time falls between the expected retention times for these two analytes, rerun the extract on a different instrument or do a post-extraction spike to confirm the identification.

To do a post-extraction spike, take a 100uL aliquot of the sample extract and add a volume of a CCV standard that will essentially double the on-column concentration of the analyte. The Benzo(b) or Benzo(k) peak that is then significantly larger than the other should be the isomer reported as present in the sample.

11.7 Method **8270D** requires verification that hits in the samples are not due to carry-over from previous runs. If a sample has a high concentration of a target compound, sufficient to saturate the detector, a clean instrument blank or sample must follow in order to report hits in any of the subsequent samples. A sample may be used in place of an IB only if the saturating target is not present at the MDL level. Narrate on the sequence which sample is being used as an IB for saturating compound(s) and reanalyze all samples that fall between the saturating run and the "blank" run.

11.8 Check the integration of the quantitation ion as you examine the spectra. Peaks should be integrated from baseline to baseline unless the matrix causes interferences with the ion ratios, in which case only the part of the peak with the correct ions should be integrated. For problematic analytes, manual integrations must be consistently applied to ICAL, CCV, and sample integrations. LIMS will place an “m”-flag next to the result for any compound that has been manually integrated and will retain both the original and the reprocessed data. If the reason for the integration is not intuitive and obvious, add a comment to the data, describing the reason for the integration.

Warning: **Manual integration performed solely to achieve calibration or other QC criteria is *illegal* and is grounds for immediate termination.**

12.0 DATA REVIEW

All data must be reviewed by a second party (peer, QC Chemist, or Department Manager) prior to reporting. See Appendix_12 for instructions on paperless procedures and working up data in LIMS. After all of the chromatograms, user reports, and final forms have been reviewed, and the data file is electronically signed by the analyst in the LIMS "Review APP", foreword the package to QC Reviewers via LIMS procedures outlined in Appendix 12.

The peer reviewer must electronically initial the user report(s) and make any additional comments on the case narrative.

13.0 POLLUTION PREVENTION

The split vent and septum purge lines are directed through a carbon trap in order to reduce solvent emissions into the laboratory.

14.0 WASTE DISPOSAL

Sample extracts should be kept in the freezer at < -10°C for at least 40 days after extraction. The extracts, and any expired standards, should then be transferred to the lab's "Chlorinated Solvents" waste stream for proper disposal.

15.0 REVISION HISTORY

The previous document (revision 12) was changed as follows:

- Cover page and numbering were reformatted
- Section 2: Updated references
- Section 3: Updated storage temperature to match current TNI/NELAP & 40CFR guidance
- Section 5: Cleaned up MDL/LOD/LOQ discussion.
- Section 9.4: Changed CCV language from "must vary" to "should vary" levels, as the "must" requirement has been dropped from the NELAP/TNI standard. Added additional notes regarding 8270D analysis.
- Appendix_2: Updated the standards information to match current usage
- Appendix_4: Updated instrument conditions to match current parameters
- Appendix_5: Minor updates to preventative maintenance instructions for clarity
- Appendix_6: Added note that 8270D lists different Tune criteria but allows use of previous 8270D criteria or other documented references.
- Appehdix_8: Removed ICAL level 10.
- Appendix_11: Revised TIC software instructions for clarity.
- Appendix_12: Revised to match current 'paperless' process.

APPENDIX_1: CALCULATIONS

SAMPLE QUANTITATION

Moisture Corrected Results for soil samples:

Dry Weight Concentration (ug/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

Concentration using Relative Response Factor (by internal standard)

Cx = [(Ax * Cis) / (Ais * RRFavg)] * pdf * idf

Where:
Cx = Concentration of the compound
Ax = Area compound
Cis = Concentration of Internal Standard, in the sample
Ais = Area of Internal Standard, in the sample
RRFavg = Average Relative Response Factor, from the curve
pdf = Prep Dilution Factor (Vf/Vi or Vf/Wi)
idf = Instrument Dilution Factor

Concentration using Linear Regression (by internal standard)

Cx = [((Ax / Ais) – b)/ m * Cis] * pdf * idf

Where:
Cx = Concentration of the compound
Ax = Area of the compound
Ais = Area of the Internal Standard
Cis = Concentration of the Internal Standard
m = slope
b = intercept
pdf = Prep Dilution Factor (Vf/Vi or Vf/Wi)
idf = Instrument Dilution Factor

Concentration via Quadratic Equation (by internal standard)

Assuming use of normal quadratic equation ($y = ax^2 + bx + c$)
Where y = Ax / Ais = Area of compound divided by Area of Internal Standard
x = Cx / Cis = Concentration of compound / Concentration of IS

$Cx = [(\sqrt{[b^2 – (4*a*(c-(Ax/Ais)))]} – b) /(2*a)]*Cis * pdf * idf$

Where:
Ax = Area of the compound
Ais = Area of the Internal Standard
Cis = Concentration of the Internal Standard
a = second order (x^2, quadratic) coefficient, from curve
b = linear (x, first order) coefficient, from curve
c = intercept, from curve
pdf = Prep Dilution Factor (Vf/Vi or Vf/Wi)
idf = Instrument Dilution Factor

This SOP contains information that may only be disseminated to C&T staff, clients & regulators.

BATCH QC

Percent Recovery (%R) is the measured concentration divided by the true concentration of the spike.

%Recovery = (SP – SA) / (C * V) *100

Where:
SP = measured concentration in the spiked sample
SA = measured concentration in the un-spiked aliquot of sample (for LCS/BS/BSD, use zero)
C = concentration of the spiking standard
V = volume used, of the spiking standard

Relative Percent Difference (RPD) is the RPD is the difference in concentrations divided by the average of the concentrations.

%RPD = (SA - DU) / ((SA + DU)/2) * 100

Where:
SA = the sample concentration
DU = the duplicate concentration.

CALIBRATION

Average Response Equations

Relative Response Factor (Rrf) = $(A_X * C_{IS}) / (A_{IS} * C_X)$

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_{IS} = Concentration of the specific internal standard
C_X = Concentration of the compound being measured

% Relative Standard Deviation (RSD) = (SD / RRFavg) * 100

Where:
SD = Standard deviation of average RF's for a compound
$= \sqrt{\{[\sum (RRFi - RRFavg)^2] / (n-1)\}}$
RRFi = Relative Response Factor for each level
RRFavg = Average Relative Response Factor from the curve
n = number of calibration points

Linear Regression Equations

y = mx + b

Where: y = response (Ax for external standard, or Ax/Ais for internal standard)
Where Ax = Area of compound
Ax / Ais = Area of compound divided by area of internal std
x = concentration (Cx for external standard, or Cx/Cis for internal standard)
m = slope

b = intercept

Slope (m) = $[(\sum wx_iy_i * \sum w) - (\sum wx_i * \sum wy_i)] / [(\sum w * \sum wx_i^2) - (\sum wx_i * \sum wx_i)]$

Intercept (b) = $y_{avg} - (m * x_{avg})$

Correlation Coefficient (or "Coefficient of Determination" for non-linear curves):

$$COD = \left(\sum_{i=1}^{n} (Yobs - Ym)^2 - ((n-1)/(n-p)) * \sum_{i=1}^{n} (Yobs - Yi)^2 \right) / \sum_{i=1}^{n} (Yobs - Ym)^2$$

Where:
- Yobs = Observed response for each concentration from each initial calibration standard, where response = Ax * (Cis/Ais)
- Ym = Mean observed response from the initial calibration
- Yi = Calculated (or predicted) response at each concentration from the initial calibration
- n = number of ICAL points
- p = Number of adjustable parameters in the polynomial equation (linear = 1, quadratic = 2)

Coefficient of Determination (r^2) = r * r

Calibration Verification (ICV & CCV)

For compounds quantitated by Average RF, calculate the CCV %Difference as follows:

%D (Percent Difference) = $((AvRF - RF_C) / AvRF) * 100$

Where:
- AvRF = Average response factor from initial calibration
- RF_C = Response factor from current daily calibration check standard

For compounds quantitated by linear/ quadratic regression, calculate the %Drift as follows:

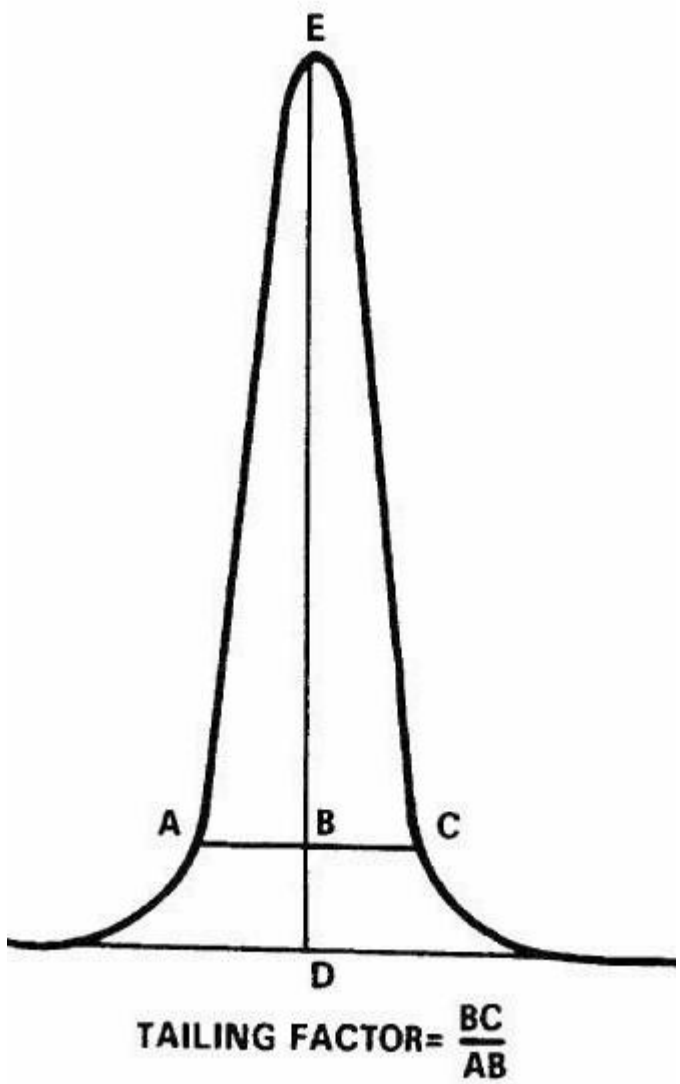
$$\%Drift = \frac{(\text{Expected Concentration} - \text{Observed Concentration})}{\text{Expected Concentration}} * 100$$

PEM (Performance Evaluation Mix)

%breakdown of DDT = $\sum (A_{DDE} + A_{DDD}) / \sum (A_{DDE} + A_{DDD} + A_{DDT})$

Where:
- A_{DDE} = Area of DDE primary ion (m/z 246)
- A_{DDD} = Area of DDD primary ion (m/z 235)
- A_{DDT} = Area of DDT primary ion (m/z 235)

Tailing Factor (from EPA 8270D):



If, using the peak at left:

Peak Height = D->E = 100 mm

10% Peak Height = B->D = 10 mm

Peak Width at 10% Peak Height = A->C = 23 mm

A->B = 11 mm
B->C = 12 mm

Tailing Factor = (B->C) / (A->B)
= 12 / 11 = 1.1

Make a Working Standard from a Source (Stock) Standard:
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where: Vss = Volume of Source Standard (mL) needed to make Working Standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

Note: If the volume to be added is less than 1µL, make an intermediate dilution by diluting the source standard 1:10 (1mL + 9 mL), then use 10 times as much of this intermediate.

APPENDIX_2: REAGENTS & STANDARDS

The standards and reagents listed below are those in use at the time this procedure was written. Alternate supplies may be used so long as they are of equivalent quality and all other calibration, quality control, and traceability requirements are met.

REAGENTS

Store reagents at room temperature.
Label each bottle with date opened/aliquotted and use or discard within one year.

Dichloromethane: EM Science, Omni-Solv grade, VWR Cat# TXDX0837-39CUT
Tuning Solution: PFTBA (perfluorotributylamine), Agilent catalog # 8500-8130

SOURCE STANDARDS

Source standards are those purchased from a chemical manufacturer or vendor. For source standards, the LIMS S-name is unique to both the composition (compound list) of the standard *and* to the vendor of that standard. A new S-name must be assigned whenever the composition is changed or when the standard is obtained from a different vendor; the information must then be entered in the "Standard Definitions" table before the new standard is assigned an S#. If you need more details, log into the LIMS browser; follow the 'LAB MENU' link and click on the "New Standards System (March 2005)" link for details on the system.

Certificates of Analysis should be obtained from the vendor of each source standard; the certificates should be labeled with the LIMS ID and the date received and filed in the 3-ring binder. Source standards should be stored protected from light in the freezer and typically have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is one year from date received. Label each vial with the contents, LIMS S#, and expiration date.

When standard ampules are opened and used, excess volume should be transferred to a 2mL screw-cap vial and retained for no more than six months. All standards must be stored in the standards freezer (at < -10°C). *Note:* Method 625 recommends storing the standards at 4°C, however the < -10°C is more conservative and results in less solvent loss.

Primary Source Standards

Analytes	Concentration (µg/mL)	Supplier & Catalog#	LIMS SS Name
DFTPP / PEM	1,000	Restek 31615	PEM/DFTPP
Internal Standards Mix	4,000	Restek 31006	SVOC IS
8270 Standard	1,000	Supelco Custom 21403668	CUSTOM8270ICV1A
8270 Standard	1,000	Supelco Custom 21403670	CSTM8270ICV2
Benzoic Acid	2,000	Supelco 47508-U	47508-U
Acidic Surrogates	10000	Restek #31087	HI_ACSURR

Base/neutral Surrogate	5000	Restek #31086	HI_BNSURR

Secondary Source Standards

Analytes	Concentration (µg/mL)	Supplier & Catalog#	LIMS SS Name
8270 Standard (MegaMix)	1,000	Restek 31850	31850
Benzidines	2,000	Restek 31834	31834
Benzoic Acid	2,000	Restek 31879	31879
Resorcinol	1,000	Restek 560299	RESORCINOL_R
Acid Surrogates	10,000	Restek 31087	HI_ACSURR
B/N Surrogates	5,000	Restek 31086	HI_BNSURR
Internal Standard Mix	4,000	Restek 31006	SVOC_IS

WORKING STANDARDS PREPARATION

Working standards are those prepared at C&T, which should be prepared in Class-A volumetric flasks. For working standards, the LIMS S-name is not unique to the source standard vendor but *is* unique to the compound list and concentrations contained in the working standard; if the concentration or compounds in the working standard changes, a new S-name, compound list and concentrations must be entered in the "Standard Definitions" table before the standard is logged in and assigned an S#. It is *very important* to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

The benchbook entry should include:

- prep date and initials of the analyst making the standard,
- LIMS S#, concentration, and volume of each Source Standard used,
- solvent name, volume, and lot#, and
- final volume, concentration, and expiration date of the new Working Standard.

Label the standards vials with the LIMS S# and the expiration date.

Working standards should be stored protected from light and expire 180 days after preparation from the source standards unless any of the source standards expire before the 180 days. If any of the source standards expire before the 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards. **The expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**. Aliquots are taken from the standard vial daily and this aliquot is used for only one day before being discarded. Store all working standards in a freezer at < -10°C; standards may not be stored in a freezer containing samples or extracts. *Note:* Method 625 recommends storing the standards at 4°C, however the < -10°C is more conservative and results in less solvent loss.

Expired standards may be used for trouble-shooting or method development so long as each standard vial is clearly marked “expired” and stored in a well-marked tray containing only expired standards.

WS Standard & Conc. (µg/mL)	Using Source Std	Add Vol (µL) Source Std	LIMS WS Name
PEM/DFTPP 50µg/L	PEM/DFTPP	1,000	DFTPP
	MeCl2	19,000	

WS Standard & Conc. (µg/mL)	Using Source Std	Add Vol (µL) Source Std	LIMS WS Name
Primary Working Standard 100/200 µg/mL	Custom8270ICVA	1,000	SCANSTOCKA
	Cstm8270ICV2	2,000	
	H1_BNSURR	100	
	H1_ACSURR	200	

WS Standard & Conc. (µg/mL)	Using Source Std	Add Vol (µL) Source Std	LIMS WS Name
ICV 40/60/100 µg/mL	31850	60	RESTEKBNAICV
	31834	45	
	31879	75	
	RESORCINOL_R	60	
	HI_ACSURR	6	
	HI_BNSURR	12	
	SVOC_IS	15	
	MeCl2	1242	

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Initial Calibration Standards

WS Standard & Conc. (µg/mL)	Using Std	Add Vol (µL) Source Std	LIMS WS Name
ICAL-1, 2/4/40/40 µg/mL	SCANSTOCKA	32	SCANICALA-1
	47508-U	32	
	SVOC_IS	16	
	MeCl2	1,536	
ICAL-2, 5/10/50/40 µg/mL	SCANSTOCKA	80	SCANICALA-2
	47508-U	40	
	SVOC_IS	16	
	MeCl2	1,480	
ICAL-3, 10/20/60/40 µg/mL	SCANSTOCKA	300	SCANICALA-3
	47508-U	90	
	SVOC_IS	30	
	MeCl2	2,610	
ICAL-4, 16/32/80/40 µg/mL	SCANSTOCKA	1,600	SCANICALA-4
	47508-U	400	
	SVOC_IS	100	
	MeCl2	8,000	
ICAL-5, 20/40/90/40 µg/mL	SCANSTOCKA	2,000	SCANICALA-5
	47508-U	450	
	SVOC_IS	100	
	MeCl2	7,550	
ICAL-6, 25/50/100/40 µg/mL	SCANSTOCKA	2,500	SCANICALA-6
	47508-U	500	
	SVOC_IS	100	
	MeCl2	7,000	
ICAL-7, 40/80/120/40 µg/mL	SCANSTOCKA	640	SCANICALA-7
	47508-U	96	
	SVOC_IS	16	
	MeCl2	864	
ICAL-8, 50/100/130/40 µg/mL	SCANSTOCKA	800	SCANICALA-8
	47508-U	104	
	SVOC IS	16	
	MeCl2	696	
ICAL-9, 60/120/140/40 µg/mL	SCANSTOCKA	960	SCANICALA-9
	47508-U	1121	
	SVOC IS	16	
	MeCl2	528	

This SOP contains information that may only be disseminated to C&T staff, clients & regulators.

APPENDIX_3: DILUTIONS

Dilutions should be prepared so that the concentration of the target analyte falls in the middle of the calibration curve.

Let the extracts warm to room temperature then:

If a 2x dilution is required, dilute a 100µL aliquot of extract with 100 µL of methylene chloride in an insert, add 2 µL of internal standard solution, and seal the vial with a crimp cap.

If the required dilution is >2x, dilute an aliquot of the extract to a total volume of 500µL in an autosampler vial, add 5µL internal standard solution and seal the vial with a crimp cap. See table below for appropriate volumes.

Shake the dilution and invert 3 times to mix.

Dilution Factor	Final Volume (µL)	Extract Volume (µL)	$MeCl_2$ Volume (µL)	IS Volume (µL)
2	200	100	100	2
5	500	100	400	5
10	500	50	450	5
20	500	25	475	5
50	500	10	490	5
100	500	5	495	5
200	500	2.5	497.5	5
250	500	2	498	5
500	500	1	499	5
1,000	1,000	1	999	10

This SOP contains information that may only be disseminated to C&T staff, clients & regulators.

APPENDIX_4: INSTRUMENT CONDITIONS

These parameters may change at the analyst's discretion to optimize instrument performance. Changes are documented in the instrument maintenance log.

BNA02:	File Designation:	**U**xxxx
	GC Model:	HP5890 Series II w/ EPC
	Autosampler:	HP7673
	Detector:	HP5971
	Injector Temperature:	270 ºC
	Detector Temperature:	300 ºC
	Oven Temperature Program:	
	Initial Temp.	40 ºC for 3.0 min
	Ramp 1	23 ºC/min to 100ºC
	Ramp 2	19 ºC/min to 280ºC
	Ramp 3	16 ºC/min to 340ºC
	Final Time	3.57 min
	Injection Volume:	1 µL
	Split-less Time:	0.75 min
	Carrier Gas:	Helium
	EPC program:	Constant flow @ 1.0 mL/min.

BNA03:	File Designation:	**V**xxxx
	GC Model:	HP6890
	Autosampler:	HP7673
	Detector:	HP5973

Injector Temperature:	270 ºC
Detector Temperature:	300 ºC
Oven Temperature Program:	
Initial Temp.	40 ºC for 3.0 min
Ramp 1	23 ºC/min to 100ºC
Ramp 2	16 ºC/min to 124ºC
Ramp 3	19 ºC/min to 340ºC
Final Time	4.0 min
Injection Volume:	1 µL
Injector:	
Mode:	Pulsed Split-less
Pressure:	16.11 psi (on)
Pulse pressure:	30.0 psi
Pulse time:	0.70 min
Purge flow:	30.0 min
Purge time	0.69 min
Total flow:	34.5 min
Carrier Gas:	Helium
PC program:	Constant flow @ 2.0 mL/min.

BNA04:	File Designation:	**W**xxxx
	GC Model:	HP5890 Series II w/ EPC
	Autosampler:	HP7673
	Detector:	HP5972
Injector Temperature:		270 ºC
Detector Temperature:		300 ºC
Oven Temperature Program:		
	Initial Temp.	40 ºC for 3.0 min
	Ramp 1	23 ºC/min to 100ºC
	Ramp 2	19 ºC/min to 280ºC
	Ramp 3	16 ºC/min to 340ºC
	Final Time	3.50 min
Injection Volume:		1 µL
Splitless time:		1.01 min
Carrier Gas:		Helium
EPC program:		Constant flow @ 1.0 mL/min.

BNA05:	File Designation:	**X**xxxx
	GC Model:	Agilent 6890N
	Autosampler:	Agilent 7683
	Detector:	Agilent 5973N

Injector Temperature:	270 ºC
Detector Temperature:	300 ºC
Oven Temperature Program:	
Initial Temp.	40 ºC for 1.5 min
Ramp 1	20 ºC/min to 290ºC
Ramp 2	2 ºC/min to 303ºC
Ramp 3	7 ºC/min to 325ºC
Final Time	0.0 min

Injector:

Mode:	Pulsed Split-less
Pressure:	17.3 psi (on)
Pulse pressure:	30.0 psi
Pulse time:	0.70 min
Purge flow:	29.4 min
Purge time	0.69 min
Total flow:	34.1 min

Column flow:

Mode:	Constant Flow
Initial Flow:	2.0 mL/min
Initial time:	0 min

BNA06:	File Designation:	**Y**xxxx
	GC Model:	Agilent 6890N
	Autosampler:	Agilent 7683
	Detector:	Agilent 5973

Injector Temperature:	270 ºC
Detector Temperature:	300 ºC
Oven Temperature Program:	
Initial Temp.	40 ºC for 1.5 min
Ramp 1	20 ºC/min to 290ºC
Ramp 2	2 ºC/min to 303ºC
Ramp 3	7 ºC/min to 330ºC
Final Time	1.64 min

Injector:

Mode:	Pulsed Split-less
Pressure:	17.3 psi (on)
Pulse pressure:	30.0 psi
Pulse time:	0.60 min
Purge flow:	29.4 min
Purge time	0.55 min
Total flow:	34.1 min

Column flow:

Mode:	Constant Flow
Initial Flow:	2.0 mL/min
Initial time:	0 min

BNA07:	File Designation:	**Z**xxxx
	GC Model:	Agilent 6890N
	Autosampler:	Agilent 7683
	Detector:	Agilent 5975
Injector Temperature:		270 ºC
Detector Temperature:		300 ºC
Oven Temperature Program:		
	Initial Temp.	40 ºC for 1.5 min
	Ramp 1	20 ºC/min to 290ºC
	Ramp 2	2 ºC/min to 303ºC
	Ramp 3	7 ºC/min to 330ºC
	Final Time	1.64 min
Injection Volume:		1 µL
Injector:		
Mode:		Pulsed Splitless
Pressure:		16.11 psi (on)
Pulse pressure:		30.0 psi
Pulse time:		0.60 min
Purge flow:		30.0 min
Purge time		0.55 min
Total flow:		34.5 min
Column flow:		
Mode:		Constant flow
Initial Flow:		2.0 mL/min
Initial time:		0 min

BNA09:	File Designation:	**R**xxxx
	GC Model:	HP7890A
	Autosampler:	HP7683B
	Detector:	HP5975C

Injector Temperature:	270 ºC
Detector Temperature:	300 ºC
Oven Temperature Program:	
Initial Temp.	40 ºC
Initial Time	1.5 min
Ramp 1	20 ºC/min to 290ºC
Ramp 2	2 ºC/min to 303ºC
Ramp 3	7 ºC/min to 321ºC
Final Time	0.929 min
Injection Volume:	1 µL

Injector:

Mode:	Pulsed Splitless
Pressure:	16.11 psi (on)
Pulse pressure:	30.0 psi
Pulse time:	0.30 min
Purge flow:	30.0 min
Purge time	0.29 min
Total flow:	34.5 min

Column flow:

Mode:	Constant flow
Initial Flow:	2.0 mL/min
Initial time:	0 min

APPENDIX_5: TROUBLE-SHOOTING & PREVENTATIVE MAINTENANCE

Instrument maintenance must be documented in the maintenance benchbook, with the exception of the daily preventative maintenance noted in the body of this SOP. If preventative or trouble-shooting maintenance is performed, document:

1.) the reason the maintenance was necessary,
2.) the action taken,
3.) the resolution of the maintenance ("passed tune", "PCP response back", etc.), and
4.) initials of the analyst performing the maintenance, and
5.) date it was performed.

File the laboratory copy of the contractor receipt in the three-ringed binder titled GC/MS-SVOC Instrument Maintenance Contractor Receipts.

PREVENTATIVE MAINTENANCE

Changing the column:
The column should be changed whenever 1.) the first analyte can not be separated from the solvent peak, 2.) an initial calibration will not meet requirements, or 3.) poor response is observed for phenols, benzidine, benzoic acid, or hexachlorocyclopentadiene, or 4.) the resolution between aniline and phenol is lost. The source should be cleaned each time the column is changed in order to minimize down-time.

1.) Use the Chemserver *INSTRUMENT CONTROL* vacuum control to vent the instrument. Then lower the GC oven temperature and turn off the injector port and detector port temperatures, and shut off the MSD diffusion pump. Unplug the rough pump.
2.) Allow all parts to cool to room temperature.
3.) Use the GC keypad to set the inlet pressure to zero and turn off the GC oven.
4.) Remove the old column from the injection and detector ports and do injector port maintenance. Cap the column with a septum.
5.) Put the injector port nut and a vespel/graphite (0.4mm ID, Restek Cat.#6717-20229) ferrule on one end of the new column, then clip about 3 or 4 centimeters off the end of the column. Hold the column at a slight downward angle when cutting the column, so that any small chips will not fall back into the column.
6.) Place a mark (with white-out or a felt-tip marker) or septa between 10-12mm from the end of the column.
7.) Insert the column into the injection port so that the lower end of the injection port nut lines up with the mark. Tighten the nut to finger-tight, then wrench-tighten until snug, making sure the mark still lines up with the end of the nut.
8.) Clip about 3 or 4 centimeters off of the detector the end of the column.
9.) Use the GC keypad to turn the carrier gas back by entering "Constant Flow" "ON".
10.) Use the GC keypad to set injector port temperature to 270°C.

11.) Set the oven temperature to 320°C and bake out the column, without installing it in the detector, while you clean the source (at least 2-3 hours but often requires 4-6 hours).
12.) After cooling the oven back to room temperature, put a new source nut (Restek Catalog # 20643) and a vespel/graphite ferrule (Restek Cat.# 20420) on the detector port end of the column. *Note:* It's very important to *not* use a pure graphite ferrule as the graphite is too soft and may crumble as the nut is tightened, releasing particles into the detector.
13.) For the 5971 and 5972 detectors, Insert the column into the detector as far as it will go, then pull it back about ¼ inch (so that tightening the nut will not crush the end of the column).
For the 5973 and 5975 detectors, leave only 2mm of the column extending from the transfer line.
14.) Tighten the nut to finger-tight, then wrench-tighten to snug.
15.) Turn on the rough pump and the MSD diffusion pump.
16.) Use the *INSTRUMENT CONTROL* options to pump down the MSD. Check the status of the instrument; once the status is "OK" (usually ~200mtorr), do a spectrum scan to check for leaks.
17.) Bake out over-night to remove any remaining moisture from the detector. The next morning, mass 28 should be less than 10%; if higher levels are observed, the injector or detector ends of the column may need to be re-installed.

Document in the maintenance log what the symptoms were that prompted you to change the column, when you changed it, and whether or not the new column eliminated the symptoms.

Cleaning the source:
The source should be cleaned whenever 1.) the column is changed, 2.) tuning criteria can not be met, or 3.) no response is obtained from either filament.

1.) Follow Steps 1 through 5 above to shut down the instrument.
2.) For 5971 and 5972 detectors, remove the column from the detector port. *Note:* The 5973 and 5975 detectors do not require this step.
3.) USE LATEX or LINT-FREE COTTON GLOVES to handle all detector parts – this is *VERY important* as the oils from your fingers will contaminate the detector.
4.) Disassemble the source as detailed in the instrument manual for that particular detector.
5.) Use the green sand-paper to remove any discoloration from the source parts, including the lenses, repeller, etc.
6.) After any discoloration has been removed, place the source parts in a methanol bath for about 20 minutes. Discard the used methanol into a flammable waste container. Repeat two more times with fresh methanol.
7.) Reassemble the source and replace both filaments, then install it onto the GC as detailed in the instrument manual.
8.) Follow Steps 16 through 22 above to reinstall the column and restart the instrument.

Document in the maintenance log what the symptoms were that prompted you to clean the source, when you cleaned it, and whether or not the cleaning eliminated the symptoms.

Trouble-shooting tip: If you do a spectrum scan and the scan shows a large primary ion of 69 with masses at 219 and 502, the tuning solution (PFTBA: perfluorotributylamine, Agilent catalog # 8500-8130) reservoir is open.

If the quadrupole rods need to be cleaned, call Full Spectrum.

Changing the rough pump oil (should be done annually):
[This is normally performed by FSA as part of the preventative maintenance contract]

1.) Follow Steps 1 through 5 above to shut down the instrument.
2.) Disconnect the hose from the rough pump.
3.) Drain the oil into the waste oil container and replace it with fresh vacuum pump oil (Scientific Instrument Supply, Catalog # INV45-1).
4.) Reconnect the hose to the rough pump and turn on the rough pump.
5.) Turn on the MSD diffusion pump, and download the detector port, injector port, and oven temperatures.
6.) Use the *INSTRUMENT CONTROL* options to check the vacuum status. Once the status is "OK" (usually ~200mtorr), do a spectrum-scan to check for hydrocarbon background; if the background is very noisy, the detector may need to be baked out.
7.) Document the maintenance in the benchbook.

TROUBLE-SHOOTING

Problem: Can't reach full vacuum

Probably a leak. Scan for m/z from 0 to 50 amu and look for water (18), nitrogen (28), and oxygen (32). If present, there's probably a leak around column-to-source connection. You can also use dust-cleaner to search for leaks by scanning for the primary ion of the main chemical in the dust-cleaner. If the abundance of that ion increases as you spray sections of the MS, then there is a leak. Check fitting and ferrule. If snug, ferrule is probably scored and should be replaced. Also check cal-gas valve.

Problem: High Background Signal

Do a spectrum scan to try to determine the source of the background signal.

- m/z 28: Nitrogen. Most likely source is a leak at the detector nut.
- m/z 31: Methanol. Were detector parts dried properly prior to installation?
- m/z 44: Carbon Dioxide. Most likely source is a leak at the detector nut.
- m/z 69 plus 219 & 502: PFTBA. The tuning solution reservoir was not closed after tuning.
- m/z 207 or 281: Siloxanes. Septum bleed or column bleed.
- m/z 446: Diffusion Pump Oil. Improper venting pulled oil into the detector. Call Agilent or Full Spectrum for assistance.
- Series of mass peaks 14amu apart, with abundance decreasing with increasing mass: Fingerprint oils on the source or detector end of the column.

APPENDIX _6: **DFTPP TUNING**

DFTPP Key Ions and Ion Abundance Criteria

Methods EPA 625 & EPA 8270C *
Semivolatile Organics by GC/MS

Mass	Ion Abundance Criteria
51	30.0% to 60.0 % of mass 198
68	< 2.0% of mass 69
69	(reference only)
70	< 2.0% of mass 69
127	40.0% to 60.0% of mass 198
197	< 1.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0% to 9.0% of mass 198
275	10.0% to 30.0% of mass 198
365	> 1.0% of mass 198
441	Present but less that mass 443
442	> 40.0% of mass 198
443	17.0% to 23.0% of mass 442

Tune acceptance is based on a single scan, average of two consecutive scans, average of three consecutive scans, or background subtraction. If the DFTPP does not pass using these options, another DFTPP should be analyzed. If that also fails, instrument maintenance should be performed to correct the problem. *No sample data associated with a failing tune standard may be reported.*

*****Note: Method 8270D lists slightly different tune criteria but allows use of other documented reference criteria including previous versions of 8270.

APPENDIX _7: CALIBRATION VERIFICATION (ICV/CCV)

An ICV (Initial Calibration Verification) standard is analyzed as part of the instrument calibration procedure described in Appendix 8 below. This standard must meet the same acceptance criteria required for the CCV (Continuing Calibration Verification) standard as described below.

A CCV (Continuing Calibration Verification) standard is analyzed after the DFTPP has passed tune, to verify that the response of the instrument has not changed significantly and that the curve may still be used to quantitate sample results. This standard must be run at the beginning of each 12-hour tune-shift, immediately after the DFTPP, using a standard at one of the three middle levels of the calibration curve, not at either extreme (highest or lowest point) of the ICAL curve. The standard concentration used for the CCV should be alternated over the course of several tune shifts.

Process the CCV through LIMS then examine the CCV summary against the following criteria to determine whether the CCV is acceptable:

i. All compounds must meet minimum relative response factor (RRF) of 0.05. See Appendix 14 for 8270D recommended minimum response factors.

j. **SPCC** (System Performance Check Compounds) RRF must be ≥ 0.05.

n-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocylclopentadiene	4-Nitrophenol

k. **CCC** (Calibration Check Compounds) percent difference (or percent drift if the compound was calibrated using linear regression) %D from the ICAL must be ≤ 20% for all DoD clients, compliance to Method 8270D allows %D ≤ 30%.

Acenaphthene	4-Chloro-3-methylphenol
Benzo(a)pyrene	2,4-Dichlorophenol
1,4-Dichlorobenzene	2-Nitrophenol
Fluoranthene	Phenol
Hexachlorobutadiene	Pentachlorophenol
N-Nitrosodiphenylamine	2,4,6-Trichlorophenol
Di-n-octyl phthalate	

l. Non-CCC compounds must have %D ≤ 30%, with the exception of poor performers (such as hexachlorocyclopentadiene, the nitroanilines, benzidine, etc.) that must have %D ≤ 40%.

Method Note: EPA 625 does not discuss minimum response requirements or differentiate between various types of analytes; the %D for each compound must be ≤ 20%.

If the SPCC and CCC criteria are met, but other compounds fail acceptance criteria, data may be reportable based on the following criteria:

a.) If the failing compound is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b.) If the compound fails the minimum RRF requirement (if RRF < 0.05) and is a required target compound for the associated samples, the samples must be reanalyzed.

c.) If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

d.) If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.

e.) If the compound fails the %D criterion due to a low response and *was* detected (even below the reporting limit), the sample must be reanalyzed.

If any of the above criteria are not met, examine the integration to verify that each peak was correctly integrated. Manual integrations must be consistently applied to ICAL, CCVs, and sample integrations. If manual integrations are performed, the file should be resent to LIMS so that a new Form 7 can be generated.

WARNING: Unsubstantiated alteration of peak integration solely to pass calibration or QC criteria is illegal and is grounds for immediate termination.

If the SPCC and CCC criteria are not met, another CCV standard should be analyzed. If two CCV's are analyzed, LIMS will use the most recent run. *Do not* "cherry pick" some compounds from the first CCV and others from the second CCV; if the second CCV is processed and used, all compounds must be taken from the second standard. If the second analysis of the standard also fails to meet the SPCC and CCC criteria, recalibration and/or other instrument maintenance is required. See Appendix_8 for the Initial Calibration procedure and acceptance criteria. See Appendix_1 for calculations.

APPENDIX_8: INITIAL CALIBRATION PROCEDURE & CRITERIA

1.0 REQUIREMENTS

An initial calibration curve must be analyzed whenever the source has been cleaned or when a new column has been installed. In general, a new calibration curve must be made whenever instrument conditions have been altered, or whenever the continuing calibration verification no longer passes acceptance criteria.

The instrument analytical range must be established by running a minimum of 5 calibration standards, containing all target compounds, at levels that bracket the quantitation range (see Appendix_2 for standard suppliers). The standards must be analyzed in order of increasing concentration. If a single standard in the curve is causing the calibration to fail, the standard may be reanalyzed, so long as it immediately follows the original curve (within the same 12 hour tune clock) and all compounds are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular compound. If a quadratic curve is used, a minimum of six points must be included in the curve.

If low detection limits are required, a sixth point should be analyzed at or below the reporting limit. This low point may be rejected only for compounds that do not require low reporting limits. The high point may be rejected for certain compounds as long as there is still a minimum of 5 points for average response or linear regression, or 6 points for quadratic regression, for each compound in the ICAL.

The curve must be verified by analyzing an Initial Calibration Verification (ICV) standard comprised of standards obtained from a different manufacturer than those used to prepare the ICAL standards. An acceptable ICV must be analyzed before any sample extracts are analyzed.

See Appendix_1 for calculation of response factors, RSD, and correlation coefficient.

2.0 PROCEDURE

2.1 Prepare the standards as described in Appendix_2.

2.2 Prepare an Initial Calibration Verification (ICV) standard from source standards obtained from a different manufacturer than the ICAL standards.

2.3 Analyze a DFTPP Tune Standard. This standard must pass acceptance criteria before the analysis can continue.

2.4 Load the calibration standards onto the autosampler tray in order of increasing concentration, adding instrument blanks before and after the ICAL standards. Load the ICV after the instrument blank that follows the calibration standards.

Note: An acceptable ICV must be analyzed before any samples are loaded. If the ICV does not pass acceptance criteria and samples were analyzed immediately following it, the entire calibration must be reanalyzed, as there is no way of determining what affect the sample matrix would have on any subsequent ICV analysis.

2.5 Load the standards in order of increasing concentration.

2.6 Use the same acquisition and processing method as used for samples (ie: 6PTBNA#.m, where "#" is the number of the instrument) but first edit the method so that all compounds are using the average response factor for quantitation and "zero" the calibration.

2.7 Write the sequence as below, identifying the type of sample as initial calibration standards, the LIMS identification of the standards, and the applicable dilution factors. This data must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information and should be written into the sequence as follows:

DFTPP, S#, 50ng
IB
ICAL, S_a#,
ICAL, S_a#,
ICAL, S_a#,
ICAL, S_a#,
ICAL, S_a#,
ICAL, S_a#,
ICAL, S_a#,
IB
ICV, S_a#,

Where:
Conc is the concentration of the standard (ie: 50 µg/mL)
S# is the LIMS S# of the standard used

3.0 ICAL ACCEPTANCE CRITERIA & DATA REVIEW

After the standards have run, examine the data to determine if the curve passes acceptance criteria.

3.1 Process any files that have not yet been processed, selecting the appropriate method file (ie: 6PTBNA#.m, where "#" is the number of the instrument), toggle each file as a calibration standard, and type the level of the standard in the space provided.

Cal Level	Concentration (µg/mL)		
1	2	4	40
2	5	10	50
3	10	20	60
4	16	32	80
5	20	40	90
6	25	50	100
7	40	80	120
8	50	100	130
9	60	120	140

3.2 Verify that each compound was detected, identified, and integrated correctly in each of the standards. Examine the retention times closely to make sure that the same peak was not identified as two analytes, particularly for isomers or other closely eluting compounds.

The retention times should increase with assigned 'compound number' (in the acquisition software); do not 're-number' the compounds, but recheck the spectra for any compound that appears to be out of order. If you must re-number, print out the spectra for the compounds that switched and have them reviewed, to confirm that the correct peak was identified.

3.3 Verify that the quantitation ions are integrated correctly. Peaks should be integrated from baseline to baseline unless the ion ratios are not correct, in which case only the part of the peak with the correct ions is integrated. Manual integrations of any kind must be substantiated and documented on the Initial Calibration Report. Manual integrations must be consistently applied to ICAL, CCV, and sample integrations. **Unsubstantiated alteration of peak integration solely to pass calibration criteria is illegal and is grounds for immediate termination.** LIMS will place an "m"-flag next to any compound that was manually integrated. If the reason for the integration is not intuitive and obvious, add a comment documenting the reason for the integration. The initial calibration curve generation and/or instrument maintenance must be repeated until specifications have been achieved.

3.4 Review the ICAL summary to determine whether or not the calibration curve complies with the following acceptance limits:

a. All compounds must meet minimum relative response factor (RRF) of 0.05 to report by 8270C; for 8270D, see Appendix 14 for minimum response criteria.

b. System Performance Check Compounds (**SPCC**) RRF must be $\geq$ 0.05:

n-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocylclopentadiene	4-Nitrophenol

c. Calibration Check Compounds (**CCC**) %RSD must be ≤ 30% for:

Acenaphthene	4-Chloro-3-methylphenol
Benzo(a)pyrene	2,4-Dichlorophenol
1,4-Dichlorobenzene	2-Nitrophenol
Fluoranthene	Phenol
Hexachlorobutadiene	Pentachlorophenol
N-Nitrosodiphenylamine	2,4,6-Trichlorophenol
Di-n-octyl phthalate	

If the RSD is > 30%, the calibration is not acceptable. If the RSD is ≥ 15%, linear regression must be used for quantitation. Correlation coefficient r2 ≥ 0.995

d. For non-CCC compounds %RSD must be ≤ 30%, with the exception of poor performers (such as hexachlorocyclopentadiene, the nitroanilines, benzidine, etc.) that must have %RSD ≤ 40%. DoD specs may be tighter.

To use the average response factor for quantitation, the non-CCC compounds must have an initial calibration RSD ≤15%.

If the %RSD is > 15%, a linear or quadratic regression must be used for this compound and the correlation coefficient (r^2) must be 0.99 or better. Use a linear regression as a first option. If, upon visual inspection of the calibration curve, there is an obvious bias at the low end, use a quadratic curve so long as there are six or more points in the curve for that compound.

Do not use weighting, force through zero, or (0,0) as a point in the curve to eliminate the low-end bias. For whichever fit is chosen, the correlation coefficient (r^2) must meet the 0.99 criteria.

e. The %D for recalculated concentrations should be within 20% of the true concentration of the standard (8000C requirement) and MUST be within 30% of the true concentration.

The 2 µg/mL standard is only required for the PAH's, which have reporting limits of 67 µg/Kg in soil. The 10µg/mL low point may be rejected for those compounds that have reporting limits greater than 10µg/mL for water or greater than 330µg/Kg in soil. The high point may be rejected for compounds that tend to saturate at high levels so long as there are at least 5 points remaining for each compound in the ICAL (or 6 points if using quadratic regression).

If a single point in the curve is causing the failure, the standard may be reanalyzed, so long as it immediately follows the original curve, is still within the same 12-hour tune clock, and all compounds are calibrated using the second run.

Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular compound.

3.5 Generate a summary report for the Initial Calibration Verification (ICV) standard, verify that the calibration standards were prepared correctly and to highlight any discrepancies between the primary- and second-source standards.

Verify that the ICV response factors and %D's meet CCV criteria. Compounds not meeting this requirement should not be reported from the calibration in question. (See Appendix 7 for the acceptance criteria and instructions on generating the ICV/CCV summary form).

An acceptable ICV must be analyzed before any samples are loaded. If the ICV does not pass acceptance criteria and samples were analyzed immediately following it, the entire calibration must be reanalyzed, as there is no way of determining what affect the sample matrix would have on any subsequent ICV analysis.

If two ICV's (containing the same analytes) were analyzed, LIMS will use the most recent run. *Do not* "cherry pick" some compounds from the first ICV and others from the second ICV; if the second ICV is processed and used, all compounds must be taken from the second standard.

3.6 Sign off on the calibration and inform the QC Chemist, Group Leader, or Department Manager that the calibration is ready for review. The reviewer must approve the calibration in LIMS before it can be used to process any data.

Note: Any corrections to the ICAL must be done through Chemstation, then resent to LIMS and a new ICAL# created. Any data processed with the draft ICAL would then need to be reprocessed against the corrected, new ICAL#.

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APPENDIX_9: COMPOUND LIST
(Quant Ion & Internal Standard Assignments)

Internal Standard	Related Compounds
1.) 1,4-Dichlorobenzene-d_4	compounds 1-18
19.) Naphthalene-d_8	compounds 19-35
36.) Acenaphthene-d_{10}	compounds 36-57
58.) Phenanthrene-d_{10}	compounds 58-69
70.) Chrysene-d_{12}	compounds 70-78
79.) Perylene-d_{12}	compounds 79-86

Compound	CAS Number	Primary Ion	Secondary Ion(s)	
1.) * 1,4-Dichlorobenzene-d_4	3855-82-1	152	150	115
2.) Pyridine [a]	110-86-1	79	52	
3.) N-Nitrosodimethylamine [a]	62-75-9	42 [c]	74	44
4.) $ 2-Fluorophenol	367-12-4	112	64	
5.) $ Phenol-d_6	13127-88-3	99	42	71
6.) Phenol	108-95-2	94	65	66
7.) Aniline [a]	62-53-3	93 [c]	66	65
8.) bis(2-Chloroethyl)ether	111-44-4	93 [c]	63	95
9.) 2-Chlorophenol	95-57-8	128	64	130
10.) 1,3-Dichlorobenzene	541-73-1	146	148	111
11.) 1,4-Dichlorobenzene	106-46-7	146	148	111
12.) Benzyl alcohol [a]	100-51-6	108	79	77
13.) 1,2-Dichlorobenzene	95-50-1	146	148	111
14.) 2-Methylphenol [a]	95-48-7	107	108	77
15.) bis(2-Chloroisopropyl)ether	108-60-1	45	77	121
16.) 4-Methylphenol [a]	106-44-5	107	108	77
17.) N-Nitroso-di-n-propylamine	621-64-7	70 [d]	42	101
18.) Hexachloroethane	67-72-1	117	201	199
19.) * Naphthalene-d8	1146-65-2	136	68	
20.) $ Nitrobenzene-d5	4165-60-0	82	128	54
21.) Nitrobenzene	98-95-3	77	123	65
22.) Isophorone	78-59-1	82	95	138
23.) 2-Nitrophenol	88-75-5	139	65	109
24.) 2,4-Dimethylphenol	105-67-9	107 [c,d]	122	121
25.) bis(2-Chloroethoxy)methane	111-91-1	93	95	123
26.) 2,4-Dichlorophenol	120-83-2	162	164	98
27.) Benzoic acid [a]	65-85-0	105 [c,d]	122	77
28.) 1,2,4-Trichlorobenzene	120-82-1	180	182	145
29.) Naphthalene	91-20-3	128	129	127

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Compound	CAS Number	Primary Ion	Secondary Ion(s)	
30.) 4-Chloroaniline [a]	106-47-8	127	129	65
31.) Hexachlorobutadiene	87-68-3	225	223	227
32.) Resorcinol [a]	108-46-3	110	53	82
33.) 4-Chloro-3-methylphenol	59-50-7	107 [d]	144	142
34.) 2-Methylnaphthalene [a]	91-57-6	142	141	
35.) 1-Methylnaphthalene [a,b]	90-12-0	142	141	
36.) * Acenaphthene-d10	15067-26-2	164	162	160
37.) Hexachlorocyclopentadiene [a]	77-47-4	237	235	272
38.) 2,4,6-Trichlorophenol	88-06-2	196	198	200
39.) 2,4,5-Trichlorophenol [a]	95-95-4	196	198	97
40.) $ 2-Fluorobiphenyl	321-60-8	172	171	
41.) 2-Chloronaphthalene	91-58-7	162	127	164
42.) 2-Nitroaniline [a]	88-74-4	65	92	138
43.) Dimethylphthalate	131-11-3	163	194	164
44.) Acenaphthylene	208-96-8	152	151	153
45.) 2,6-Dinitrotoluene	606-20-2	165	63	89
46.) 3-Nitroaniline [a]	99-09-2	138	108	92
47.) Acenaphthene	83-29-9	154	153	152
48.) 2,4-Dinitrophenol	51-28-5	184	63	154
49.) 4-Nitrophenol	100-02-7	109 [c,d]	139	65
50.) Dibenzofuran [a]	132-64-9	168	139	
51.) 2,4-Dinitrotoluene	121-14-2	165	63	89
52.) 2,3,4,6-Tetrachlorophenol [a]	58-90-2	232	230	234
53.) Diethylphthalate	84-66-2	149	177	150
54.) 4-Chlorophenyl-phenyl ether	7005-72-3	204	206	141
55.) Fluorene	86-73-7	166	165	167
56.) 4-Nitroaniline [a]	100-01-6	138	65	108
57.) $ 2,4,6-Tribromophenol	118-79-6	330	332	141
58.) * Phenanthrene-d10	1517-22-2	188	94	80
59.) 4,6-Dinitro-2-methylphenol	534-52-1	198	51	105
60.) N-Nitrosodiphenylamine [a]	86-30-6	169	168	167
61.) Azobenzene [a,b]	103-33-3	77	105	182
62.) 4-Bromophenyl-phenyl ether	101-55-3	248	250	141
63.) Hexachlorobenzene	118-74-1	284	142	249
64.) Pentachlorophenol	87-86-5	266	264	268
65.) Phenanthrene	85-01-8	178	179	176
66.) Anthracene	120-12-7	178	179	176
67.) Carbazole [a,b]	86-74-8	167	166	139
68.) Di-n-butylphthalate	84-74-2	149	150	104
69.) Fluoranthene	206-44-0	202	101	203

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Compound	CAS Number	Primary Ion	Secondary Ion(s)	
70.) * Chrysene-d12	1719-03-5	240	120	236
71.) Benzidine [a]	92-87-5	184	92	185
72.) Pyrene	129-00-0	202	200	203
73.) $ Terphenyl-d14	1718-51-0	244	122	212
74.) Butylbenzylphthalate	85-68-7	149	91	206
75.) 3,3'-Dichlorobenzidine	91-94-1	252	254	126
76.) Benzo(a)anthracene	56-55-3	228	229	226
77.) bis(2-Ethylhexyl) phthalate	117-81-7	149	167	279
78.) Chrysene	218-01-9	228	226	229
79.) * Perylene-d12	1520-96-3	264	260	265
80.) Di-n-octyl phthalate	117-84-0	149	167	43
81.) Benzo(b)fluoranthene	205-99-2	252	253	125
82.) Benzo(k)fluoranthene	207-08-9	252	253	125
83.) Benzo(a)pyrene	50-32-8	252	253	125
84.) Indeno(1,2,3-cd)pyrene	193-39-5	276	138	277
85.) Dibenz(a,h)anthracene	53-70-3	278	139	279
86.) Benzo(g,h,i)perylene	191-24-2	276	138	277

* Internal standard compound
$ Surrogate compound

Method Modifications:

[a] Compound is not an EPA 625 target analyte.
[b] Compound is not an EPA 8270C target analyte.
[c] Quantitation based on secondary ion with an abundance equivalent to or greater than that identified in method 8270. C&T's primary ion differs from the primary ion listed in the method due to presence of nearby compounds with similar ions.
[d] Quantitation based on secondary ion with an abundance equivalent to or greater than that identified in method 625. C&T's primary ion differs from the primary ion listed in the method due to presence of nearby compounds with similar ions.

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APPENDIX_10: C&T STANDARD REPORTING LIMITS*

CAS #	Target Compound	Reporting Limits (µg/L)	Reporting Limits (µg/Kg)
83-32-9	Acenaphthene	10	67
208-96-8	Acenaphthylene	10	67
120-12-7	Anthracene	10	67
103-33-3	Azobenzene	10	330
56-55-3	Benzo(a)anthracene	10	67
50-32-8	Benzo(a)pyrene	10	67
205-99-2	Benzo(b)fluoranthene	10	67
207-08-9	Benzo(k)fluoranthene	10	67
191-24-2	Benzo(g,h,i)perylene	10	67
65-85-0	Benzoic acid	50	1,700
100-51-6	Benzyl alcohol	10	330
111-91-1	bis(2-Chloroethoxy)methane	10	330
111-44-4	bis(2-Chloroethyl)ether	10	330
108-60-1	bis(2-Chloroisopropyl)ether	10	330
117-81-7	bis(2-Ethylhexyl)phthalate	10	330
101-55-3	4-Bromophenyl-phenylether	10	330
85-68-7	Butylbenzylphthalate	10	330
106-47-8	4-Chloroaniline	10	330
59-50-7	4-Chloro-3-methylphenol	10	330
91-58-7	2-Chloronaphthalene	10	330
95-57-8	2-Chlorophenol	10	330
7005-72-3	4-Chlorophenyl-phenylether	10	330
218-01-9	Chrysene	10	67
53-70-3	Dibenz(a,h)anthracene	10	67
132-64-9	Dibenzofuran	10	330
95-50-1	1,2-Dichlorobenzene	10	330
541-73-1	1,3-Dichlorobenzene	10	330
106-46-7	1,4-Dichlorobenzene	10	330
91-94-1	3,3'-Dichlorobenzidine	20	670
120-83-2	2,4-Dichlorophenol	10	330
84-66-2	Diethylphthalate	10	330
105-67-9	2,4-Dimethylphenol	10	330
131-11-3	Dimethylphthalate	10	330
84-74-2	Di-n-butylphthalate	10	330
534-52-1	4,6-Dinitro-2-methylphenol	20	670
51-28-5	2,4-Dinitrophenol	20	670
121-14-2	2,4-Dinitrotoluene	10	330
606-20-2	2,6-Dinitrotoluene	10	330

CAS #	Target Compound	Reporting Limits (µg/L)	Reporting Limits (µg/Kg)
117-84-0	Di-n-octylphthalate	10	330
206-44-0	Fluoranthene	10	67
86-73-7	Fluorene	10	67
118-74-1	Hexachlorobenzene	10	330
87-68-3	Hexachlorobutadiene	10	330
77-47-4	Hexachlorocyclopentadiene	20	670
67-72-1	Hexachloroethane	10	330
193-39-5	Indeno(1,2,3-cd)pyrene	10	67
78-59-1	Isophorone	10	330
91-57-6	2-Methylnaphthalene	10	330
95-48-7	2-Methylphenol	10	330
1319-77-3	4-Methylphenol	10	330
88-74-4	2-Nitroaniline	20	670
99-09-2	3-Nitroaniline	20	670
100-01-6	4-Nitroaniline	20	670
88-75-5	2-Nitrophenol	20	670
100-02-7	4-Nitrophenol	20	670
621-64-7	N-Nitroso-di-n-propylamine	10	330
62-75-9	N-Nitrosodimethylamine	10	330
86-30-6	N-Nitrosodiphenylamine	10	330
91-20-3	Naphthalene	10	67
98-95-3	Nitrobenzene	10	330
87-86-5	Pentachlorophenol	20	670
85-01-8	Phenanthrene	10	67
108-95-2	Phenol	10	330
129-00-0	Pyrene	10	67
120-82-1	1,2,4-Trichlorobenzene	10	330
95-95-4	2,4,5-Trichlorophenol	10	330
88-06-2	2,4,6-Trichlorophenol	10	330
Additional Compounds (may be added to list):			
62-53-3	Aniline	50	1,700
103-33-3	Azobenzene	10	330
92-87-5	Benzidine	50	1670
86-74-8	Carbazole	10	330
90-12-0	1-Methylnaphthalene	10	330
110-86-1	Pyridine	100	1,700
108-46-3	Resorcinol	20	670
58-90-2	2,3,4,6-Tetrachlorophenol	50	1,700

*The reporting limits listed above are based on the extraction of 1L of water or 30g of soil, with a final extract volume of 1.0mL.

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APPENDIX_11: TENTATIVELY IDENTIFIED COMPOUNDS (Library Searches)

A "Library Search" can be used to identify and estimate the concentration of non-target analytes that may be present in the sample. The software will compare the spectra of the unknown peaks to those in the NIST (National Institute of Standards & Technology) database, which contains approximately 70,000 compounds. This comparison is done by matching the relative abundance of the unknown to that of the library compound using Probability Based Matching (PBM), and then producing a Tentatively Identified Compound (TIC) list. Clients may request library searches by referencing the "PBM" or "TIC" acronym.

Library Searches are done and print automatically if the library search toggle is on. When reporting TICS, C&T reports up to 10 peaks. If there are more than 10 peaks, report the 10 with the greatest area. Any peak area for which the area is less than 10% of the corresponding ISTD will not be reported. Methods 625 and 8270C/D don't specify how may TIC's to report, client specifications may require reporting more than 10 TIC's.

Note: Sulfur may appear as a very wide (~5minutes total) peak with ions of mass 64, 96, 128, 160, 192, and 256. Add a note to the job stating which samples it appeared in and if dilutions were required to avoid it's interference, but don't report it as a TIC, as it is not an organic compound and there are wet chemistry methods for analysis of sulfur and sulfide.

1.0 Finding the Unknown Compounds

Chemstation can perform a Library Search to look for unknowns and produce a Tentatively Identified Compound (TIC) quantitation report. However, any unknown peaks that fall within a target analyte retention time window will not be found by the automatic search. Therefore, we must examine the quant results to check for missed unknowns, then QEdit when necessary. Make sure to review all data files before doing the TICs so the correct data will be sent to LIMS. Use the following steps to perform the search:

1.1 In the Chemstation Browser, right-click on the datafile and select "Load".

1.2 Under "Quant", click "QEdit Quant Results" and click through each analyte.

1.3 If there was a mis-identified target compound, click "QDel" and this will change the status from target to unknown.

1.4 Once done, click "Exit" and save the changes.

1.5 In the Chemstation browser, under 'LSC', click 'LSC Summary to Screen' and click 'yes' to continue. This will bring up each unknown with up to 5 possible library search matches.

The first potential match will be that with the highest quality match but if the sample spectrum does not match the reference spectrum, you can select from one of the other four.

If none of the potential matches resembles the sample spectrum, that peak should be identified as an "unknown". Any peak with less than a 50% match will automatically be tagged as 'unknown'.

1.6 Click 'Done when finished.

1.7 In the Chemstation browser, under "LSC", click on 'LSC Detailed to Printer" to sent the TIC report to LIMS.

1.8 Go to LIMS and in the Review-App, go to the sample data file and click "TIC" to edit. This is where you can change hits to 'Unknown".

2.0 Identification of Non-Target Compounds (Library Searches)

If less than 10 unknowns are found, work up all the unknown compounds. If greater than 10 unknowns are found, LIMS will automatically select the top ten unknown peaks by area, where the area is greater than 1/10 the area of the nearest internal standard. When computer searches are performed, visual verification of the computer match is required along with meeting the following guidelines:

2.1 All ions of greater than 10% relative intensity in the library spectrum should be present in the sample.

2.2 Relative intensities of the ions must agree to within plus or minus 20% between library spectrum and sample spectrum.

2.3 Molecular ions in the library spectrum must be in the sample spectrum.

2.4 Ions present in the sample spectrum but not in the library spectrum should be checked for co-elution of other compounds and considered for background subtraction.

Note: The detectors are set to begin scanning at m/z 35. Therefore, masses less than 35 (in the reference spectra) should not be used to identify the unknowns.

2.5 Verify that the system has not identified an unknown peak as a target compound, which it may do if the spectrum of the unknown is very similar to that of a target compound. If it did, review the pattern and %match for the second most-probable match and if that match is at all acceptable, report the second compound.

3.0 Interferences

If there is a leak at the detector nut, Carbon Dioxide (ion m/z 44) and/or Argon (ion m/z 40) may be found in the library search. These masses should not be considered when evaluating the sample spectrum against the possible matches.

3.1 Siloxanes are byproducts of column degradation and should not be reported as TIC's.

3.2 Review and report TICs found in the Method Blank associated with the sample(s) to ensure any byproducts of the extraction process are documented as such. Any TICs identified in the Method blank are to be considered a product of the extraction process, and the same TIC found in the associated sample(s) should be narrated as a product of the extraction process, not native to the sample itself.

APPENDIX_12: WORK UP DATA IN LIMS

Data workup in LIMS is accomplished through the “Review Application”.

A) Sequence Workup

1.) Go to your day’s Sequence in LIMS. In the top right, use the "Review" pull down menu and select your Sequence (for example: MSBNA09 10/18/12).

2.) Login if necessary.

3.) Check the tune and if it passes, sign off on your tune run.

4.) Check the CCV integrations. If any changes are needed:

- 4.1) Make the changes in the original data file and save the file.
- 4.2) Upload the corrected file to LIMS.
- 4.3) Click the Refresh button or Click on the same sample row in the Review App. to see the corrected files.
- 4.4) Narrate all manual integrations and their reason.
- 4.5) Check and sign off on the CCV.

5.) Go down the sample list, by first clicking on the row of the Blank run.

6.) Check IS and Surrogate recoveries for any failures on the LIMS Report.

7.) Check the Total Ion Chromatogram and the Report from the LIMS-captured report for any high non-target or interfering peaks, or any peaks missed due to excessively high concentration.

8.) Go through each analyte:

- 8.1) Check if the calculated value is a "hit".
- 8.2) Check the integration to see if it is correct. If it is not, go back to the original data file and make the manual integration. Save your change and upload the file to LIMS. Hit the Refresh button in the Review App. and confirm and narrate the change is in LIMS.
- 8.3) Compare the spectra to determine if the selected analyte is indeed the target analyte.
- 8.4) Make any changes to the flags for that analyte (ie. false positive)

9.) Add any necessary comments for the run. Provide the dilution factor if a re-run is needed or other relevant comments.

10.) Click Report to choose the analytes from that run. This will mark every analyte as usable (with a “u” flag), except for those that are >LR.

11.) If necessary, click ‘Re-Run’ to flag the run for re-analysis. This will not remove your "u"-flags for the analytes that you wish to report.

12.) Once your comments have been entered and your analytes are chosen, Click ‘Sign’ to sign off on the run.

13.) Click on the next run or ‘Save + Next’ to go to the next run.

14.) Once all samples for the sequence have been worked up, click on the Sequence in the top left corner of the Review App and verify the sequence.

B) Batch Workup

1.) Go to the daily sequence in LIM. In the top right corner of the screen, use the ‘”Review” pull down menu and select the batch that needs review.

2.) Login if necessary.

3.) Go through each QC run and check for two things:

3.1) first, check that the “general version” of that QC run is reportable. If not, then the batch must be reanalyzed.
3.2) Second, check if that QC passed the “client-specific” limits. If not, then must be reanalyzed.

Note: See Section 10.5 ‘Batch QC’ above for guidance on the appropriate corrective action for batch QC failures.

4.) If both conditions are met, review the prep log paperwork and then sign off on the batch and send it for secondary review.

C) Job Workup

1) Go to your daily Sequence in LIMS. In the top right, use the "Review" pull down menu and select the job you would like to report.

2) Login if necessary.

3) Go through each sample and check that any requested analytes are chosen ("u"'ed) and each run reporting an analyte is signed. If some of the samples run were worked up by another analyst, use this opportunity to peer review those runs.

4) Then go through each QC run and check that the batch QC passed any client-specific limits.

5) If QC limits were met, click Sign to sign off on the specific QC results. If the general version for a given QC run has not been signed, go into the Sequence in the Review App and review the raw data for that QC.

6) Once all samples and QC have been reviewed and signed, select the pull-down menu that says "all" and select "pkg".

7) Click ‘Reports’ then choose which Form 1's to print. Click the Refresh button at the top of the Report Manager until the report lines change colors.

8) Next click ‘Done’. The Form 1's will now appear on the right in the Review App.

9) Click on Checklist to review all criteria, ensure all appropriate comments are added to the narrative.

10) Make any necessary comments for the Job in the Review App.

11) Click Sign to sign off on the Job and send for review.

12) If the Job is Level III or Level IV, then the Peer Reviewer will be responsible for Generating and Signing off on the Level III or Level IV part of the job.

D) Peer Review

1) Open the job from Analyst review queue.

2) Login if necessary. Then you'll be in PKG mode.

3) Go through each sample and review the raw data that was captured.

   - Check the Total Ion Chromatogram for any peaks missed due to excessive high concentrations or interferences.
   - Check the dilution factor for the sample.
   - Check that the analytes are flagged and “u”-ed properly.
   - Verify that any relevant comments have been added.
   - Sign the sample run.

4) After all the samples are second-signed, go through the QC and check for two signoffs for the general versions. Then review the user reports for the specific versions and sign off on them.

5) Next check that all sequences, ICAL, Tunes, and CCVs reported in this job have two signoffs (aka "SR").

6) Compare the paper Form 1's with the user reports in LIMS to check for errors.

7) If the job is Level II, sign off on the job.

8) If the job is Level III or IV, click on the Level III or Level IV row and generate a Level III or IV. You will then see a log that will indicate what else needs review before the Level III or IV is ready to report. Investigate, fix, and re-generate the Level III or IV. Again check the log. When the log only states at what time/date the Level III or Level IV was generated, sign off on the Level III or IV. Then sign off on the job.

E) ICAL (Initial Calibration) Workup

1) After the ICAL has run, verify that the documents for the ICAL/ICV have been captured in LIMS. All samples that have a document captured will have a "D" to the right of the sample type in the LIMS sequence screen.

2) Check the LIMS ICAL summary and the ICAL error report for any problems. This report will display calculation mismatch errors between Chemstation and LIMS, and bad linear or quadratic curves that quantitate the lower point below our in-house threshold.

3) If there are problems displayed on the ICAL error report, investigate and correct the method in the data acquisition/processing software.

4) Take some time to check any linear or quadratic curves using the "Edit" feature in LIMS. You might be able to create a better curve fit, by dropping high points or change the curve type. The "Edit" feature is to be used as a tool to help determine the best curve fit for a given analyte. Therefore, you *must* apply any and all changes to the processing method. In addition, a new initial calibration *must* be created in LIMS. Check the report for the results of the changes made.

5) Review the requantitation of all calibration points to ensure they are no more than +/-30% of the actual concentration. If the curve is biasing the quantitation of the compound the curve may need adjustment.

6) Carefully inspect the high end of the calibration range, both chromatographically and qualitatively. If there are signs of saturation towards the high end indicated by either a sharp decline in the requantitation of a compound, or a fronting/broadened peak, drop that point from the calibration. Leaving saturated points in a calibration curve can introduce a low bias, especially in high concentration hits.

7) Again check for any errors. If there are, Repeat step 4 above until the ICAL error report is free of problems.

8) Review any manual integration that was needed for the ICV.

9) Narrate all manual integrations and/or changes for each ICAL level.

10) Sign off on all data files, sequence, and the initial calibration.

APPENDIX_13: EPA 625

The following criteria and discussion apply to any samples submitted for the EPA 625 analysis:

Tuning (DFTPP) & Performance Evaluation Mix (PEM):
The tailing factors specified in 625 match those in 8270 and C&T criteria; see the PEM section in the main body of the SOP for additional discussion and criteria. The 50ng DFTPP acceptance criteria listed in EPA 625 are the same as the EPA 8270 criteria and can be found in Appendix_6.

Method Modification: EPA 625 does not require the inclusion of DDT in the PEM and so does not include breakdown criteria.

Initial Calibration:
Method 625 only requires 3 points and average response can be used so long as the RSD is ≤35%; a regression curve may be used if the RSD exceeds 35% or whenever desired. Although 625 only requires 3 calibration points, the same general calibration criteria must be followed (ie: intermediate levels may not be dropped). Any calibrations to be used for both 8270 and 625 must meet the SW-846 requirements described in Appendix_8 above. See the procedure described in Appendix_8 above for details.

Calibration Verification (ICV/CCV):
Method 625 does not discuss the use of an ICV (Initial Calibration Verification standard) to validate the initial calibration curve, however C&T's NELAC accreditation requires it; the ICV must be analyzed and approved following the procedure described in Appendix_7 above.

Method 625 also only requires that a CCV (Continuing Calibration Verification) standard be analyzed once daily, however C&T policy is to follow the SW-846 guidance and analyze the CCV at the beginning of each 12-hour tune-shift. The %D for each analyte must be ≤ 20%; if it does not, use the corrective action guidance described in Appendix_7 to determine the appropriate course of action.

Batch QC:
EPA 625 defines the LCS and MS recovery limits in Table 6 of the method, however C&T statistically generated limits are consistently tighter than those identified in this table; C&T uses the tighter, statistically generated limits.

Instrument Conditions:
Curtis & Tompkins uses current technology, namely capillary columns in place of the packed columns described in the method, so instrument conditions are somewhat different than those listed in the method. See Appendix_4 and instrument maintenance logs for instrument parameters.

Quantitation:
The ions used for quantitation are listed in Appendix 9, including identification of those whose quantitation is based on a secondary ion.

APPENDIX_14: 8270D MINIMUM RRF CRITERIA

If samples are being analyzed for EPA 8270D (Feb.2007), the ICV and CCV must meet the following minimum relative response factors (RRF). Those not specified in the list below should meet C&T criteria of 0.05.

N-Nitrosodimethylamine	-	2,4-Dinitrophenol	0.010
Pyridine	-	4-Nitrophenol	0.010
Phenol	0.800	Dibenzofuran	0.800
Aniline	-	2,4-Dinitrotoluene	0.200
Bis(2-Chloroethyl)ether	0.700	2.3.4.6-Tetrachlorophenol	0.010
2-Chlorophenol	0.800	Diethylphthalate	0.200
1,3-Dichlorobenzene	-	Fluorene	0.900
1,4-Dichlorobenzene	-	4-Chlorophenyl-phenyl-ether	0.400
Benzyl alcohol	-	4-Nitroaniline	0.010
1,2-Dichlorobenzene	-	4,6-Dinitro-2-methylphenol	0.010
2-Methylphenol	0.700	N-Nitrosodiphenylamine	0.010
Bis(2-Chloroisopropyl)ether	0.010	Azobenzene	-
4-Methylphenol	0.600	4-Bromophenyl-phenylether	0.100
N-Nitroso-di-n-propylamine	0.500	Hexachlorobenzene	0.100
Hexachloroethane	0.300	Pentachlorophenol	0.050
Nitrobenzene	0.200	Phenanthrene	0.700
Isophorone	0.400	Anthracene	0.700
2-Nitrophenol	0.100	Carbazole	0.010
2,4-Dimethylphenol	0.200	Di-n-butylphthalate	0.010
Bis(2-Chloroethoxy)methane	0.300	Fluroanthene	0.600
Benzoic Acid	-	Benzidine	-
2,4-Dichlorophenol	0.200	Pyrene	0.600
1,2,4-Trichlorobenzene	-	Butylbenzylphthalate	0.010
Naphthalene	0.700	3,3'-Dichlorobenzidine	0.010
4-Chloroaniline	0.010	Benzo(a)anthracene	0.800
Hexachlorobutadiene	0.010	Bis(2-Ethylhexyl)phthalate	0.010
4-Chloro-3-methylphenol	0.200	Chrysene	0.700
2-Methylnaphthalene	0.400	Di-n-octylphthalate	0.010
1-Methylnaphthalene	-	Benzo(b)fluoranthene	0.700
Resorcinol	-	Benzo(b)fluoranthene	0.700
Hexachlorocyclopentadiene	0.050	Benzo(a)pyrene	0.700
2,4,6-Trichlorophenol	0.200	Indeno(1,2,3-cd)pyrene	0.500
2,4,5-Trichlorophenol	0.200	Dibenz(a,h)anthracene	0.400
2-Chloronaphthalene	0.800	Benzo(g,h,i)perylene	0.500
2-Nitroaniline	0.010	2-Fluorophenol	-
Dimethylphthalate	0.010	Phenol-d5	-
2,6-Dinitrotoluene	0.200	Nitrobenzene-d5	-
Acenaphthylene	0.900	2-Fluorobiphenyl	-
3-Nitroaniline	0.010	2,4,6-Tribromophenol	-
Acenaphthene	0.900	Terphenyl-d14	-

Curtis & Tompkins, Ltd.

DIGESTION & ANALYSIS OF SOLID SAMPLES FOR

MERCURY ANALYSIS

EPA 7471

Approved by:

Porter Williams 3/9/12
Department Manager/ Date

3/9/12
QA Director/ Date

Read & Understood & Agreed by:

3.09.12
Signature/ Date

Signature/ Date

3/9/12
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Re-Approved by:

2/25/13
Signature/ Date

Signature/ Date

This SOP contains information that may only be disseminated to C&T staff, clients & regulators.

Curtis & Tompkins, Ltd.

Mercury Analysis of Solid Samples
by Cold Vapor AA Method EPA 7471

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This SOP contains information that may only be disseminated to C&T staff, clients & regulators.

Curtis & Tompkins, Ltd.

Mercury Analysis of Solid Samples by Cold Vapor AA

A. SCOPE

This procedure describes the sample preparation and analysis for determining the concentration of mercury in soils, sediments, bottom deposits, and sludge-type materials. The reporting limit for this procedure, using a 0.6g sample, is 0.02 mg/Kg.

In this procedure, organometallic compounds are broken down and converted to mercuric ions by digesting the sample with permanganate and persulfate under heated conditions. The concentration of mercury in the resulting digestate is then determined by cold vapor atomic absorption (CVAA). This procedure is not applicable to Waters or Liquid Samples (see Metals SOP Section 5.1, Method 7470A).

B. REFERENCES

EPA 7471A, Mercury in Solid or Semisolid Waste *(Manual Cold-Vapor Technique)*, SW846 Update 1, Sept 1994
EPA 7471B, Mercury in Solid or Semisolid Waste *(Manual Cold-Vapor Technique)*, SW846 Update 2, Feb 2007
Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Rev 4.2 October 2010
Additional SOP's and Guidance Documents:
EPA 7000A, *Atomic Absorption Methods*, SW-846 Update 3, Feb 2007
NELAC Chapter 5 Quality Systems, 6/5/2003, Sec 5.5.4-5.5.6
DoD Quality Systems Manual, Version 4.1, 4/22/09
DoE Quality Systems Manual, Version 2.2, October 2006
C&T SOP QA 1.4, *Balance Calibration Check & Maintenance*
C&T SOP QA 1.5, *Calibrating & Maintaining Temperature Controls*
C&T SOP QA 1.6, *Pipet Calibration Check Procedures*
C&T SOP QA 1.9, *Calibration of Volumetric Glassware*
C&T SOP QA 1.5, *Calibrating Thermometers and Maintaining Temperature Controls*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL)*

C. SAMPLE PRESERVATION & HOLDING TIME

Preservative: No chemical preservation. Store at 4°C.
Holding time: 28 days

D. SAFETY

Mercury is a highly toxic element that is also somewhat volatile. The reagents used in this procedure are corrosive and can be harmful if inhaled. Always perform the digestions under a hood and take steps to avoid inhaling vapors generated in the process.

This procedure uses concentrated acids that will cause injury if allowed to contact skin or eyes.

Gloves and safety glasses and appropriate clothing should be worn at all times when handling samples, standards, or reagents. Aqua regia must be used in the fume hood due to toxic and corrosive fumes.

E. INTERFERENCES

Mercury is a relatively volatile element that may be vaporized if the digestion temperature is not closely monitored. Sulfide interferences are reduced through use of potassium permanganate in the digestion step. Samples with high organic content may reduce the potassium permanganate and require additional permanganate be added during digestion.

F. QC REQUIREMENTS

Initial Calibration curves must be composed of a minimum of a blank and five standards, which are digested in the same manner as the associated samples. Instrument should be calibrated before each analytical sequence. The correlation coefficient must be $\geq$ 0.995.

Initial Calibration Verification (ICV) standards must be prepared from stock standards received from a source other than those used for the initial calibration curve. The ICV is analyzed prior to sample analysis with a recovery between 90-110%. (%D = +/- 10%)

Initial Calibration Blank (ICB) is a reagent blank that follows the ICV. Mercury must not be detected above one half (1/2 or 50%) of the Reporting Limit (RL).

Continuing Calibration Verification (CCV) standards must be analyzed after every 10 samples, including batch QC samples, and at the end of the analytical sequence. Since the instrument is calibrated every day, the concentration of the CCV standards is analyzed in the mid-range of the calibration curve. The recovery for the CCV must be within 80-120%. If a CCV fails to meet the recovery criteria, the analysis is terminated, the instrument re-calibrated, and all samples analyzed after the last passing CCV must be reanalyzed.

Calibration Blanks (CCB) are reagent blanks that follow every CCV. Mercury must not be detected above the MDL.

Batch QC: The following quality control (QC) samples must be prepared in the same manner as the analytical samples at a rate of once per twenty or less samples. C&T in-house limits are based on control charts and are updated semi-annually; see the associated SOP *Mercury QC Limits, Table-1* for the current limits.

Method Blank (MB): Digest and analyze a method (prep) blank with each batch of 20 samples or less to demonstrate that the glassware and reagents are free of contamination. Mercury must not be detected above one half (1/2 or 50%) of the Reporting Limit (RL)

Blank Spike / Blank Spike Duplicate (BS/BSD): Digest and analyze a blank spike and a blank spike duplicate for every batch of 20 samples or less to demonstrate that the

procedure is accurate (measured by the *recovery*) and precise (measured by the *RPD*) in the *absence* of matrix interferences.

Matrix Spike (MS) / Matrix Spike Duplicate (MSD): Digest and analyze an MS/MSD with every batch of samples to demonstrate the accuracy and precision of the process on real-world samples.

Note: Client- or project-specific quality assurance project plans (QAPPs) may require an MS/MSD in place of the SDUP and SSPIKE. For batches containing samples from these projects, the QAPP requirement takes precedence over this SOP.

Sample Interference Verifications: The method recommends that whenever a new or unusual matrix is encountered, a series of tests be performed to ensure that neither positive nor negative interferences are distorting the accuracy of the reported values. These are optional steps that should be implemented for any batch containing "Level 3" or Level 4" samples, as nearly all Department of Defense project plans include this requirement. These should also be analyzed whenever the analyst suspects that sample viscosity, salt content, or other matrix interferences are likely.

Serial Dilutions: Analysis of a 5x dilution should agree within ± 10% (90-110% recovery) of the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference should be suspected.

Post Digest Spikes: An analyte spike added to a portion of a prepared sample digest, or its dilution, should be recovered to within 75-125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits a matrix effect should be suspected.

Method Detection Limit (MDL): MDL studies are performed when major changes to instruments or methods occur, in order to demonstrate that the sample preparation and analysis procedures are adequate to meet required reporting limits. The MDL is determined by digesting and analyzing at least 7 replicates of a low-level blank spike, determining the standard deviation for each element, and applying a multiplier. See the QA SOP "Determining MDLs" for details.

Limit of Detection (LOD) must be determined quarterly for DoD and annually for NLEAC compliance and is based on a laboratory control sample (MDL check sample) that is spiked 2 to 4 times the MDL, digested and analyzed on every instrument. See the QA SOP for Method Detection Limits for details.

Limit of Quantitation (LOQ) must be determined quarterly for DoD and annually for NELAC and is based on a laboratory control sample that is spiked 1 to 2 times the reporting limit. It is only analyzed once per method. See the QA SOP for Limit of Quantitation for

details.

Instrument Detection Limit (IDL): The IDL is determined by analyzing seven instrument blanks on three non-consecutive days. The standard deviation of the calculated concentrations is determined for each day; the IDL is the sum of the three standard deviations.

Glassware: Class-A digestion tubes should be purchased, or the calibration of the disposable digestion tubes must be verified, to within 3%, for each lot received. Per the SOP for Calibrating Volumetric Glassware.

G. EQUIPMENT (See Appendix_5 for instrument conditions)

Heating block capable of maintaining 90-95 °C for 30 minutes. Calibrated annually along with the thermometers for temperature control within +/- 2C by SOP for Temperature Controls
LEEMAN "Hydra AA" automated mercury analyzer
Disposable 70 mL digestion tubes, CPI Catalog # 4370-010010
0.22 µm Syringe Filters, Millipore Catalog # SLGV033NK

Note: If the digestion tubes are not certified by the manufacturer or vendor as Class-A, the accuracy of each lot must be verified prior to use according to SOP for Volumetric Labware. This is done by selecting 5 digestion tubes from the lot and filling them to 50mL with 20°C deionized water and weighing it on an analytical (4 decimal) balance; the weights must fall within 3% of the expected weights (48.5 – 51.5g) or the lot may not be used for direct measurement of sample or digestate volume.

H. PREPARE THE CALIBRATION STANDARDS

Initial calibration standards: Add the following volumes of a 0.100 µg/mL mercury working standard to a series of digestion tubes then follow the digestion procedure below, starting at Step-13 so the volume of the standards matches that of the samples. The LIMS ID of the 100 µg/mL working standard must be documented in the digestion log. These standards expire after 24 hours.

Cal Level	Final Concentration (ug/L)	Volume of 0.100µg/mL Working Std (mL)	Final Volume (mL)
Cal Zero	0.0	0.00	50
Std #1	0.2	0.10	50
Std #2	0.5	0.25	50
Std #3	2.0	1.00	50
Std #4	5.0	2.50	50
Std #5	10.	5.00	50

ICV and CCVs: The Initial Calibration Verification (ICV) standard must be made from intermediates obtained from a different manufacturer than those used to make the initial calibration curve and the Continuing Calibration Verification (CCV) standards may be

prepared from either of the sources. *Note:* The US Army Corp of Engineers recommends that the CCV's be prepared from the same source standards as the ICAL curve. An acceptable ICV must be analyzed before any samples are loaded. If the ICV does not pass acceptance criteria and samples were analyzed immediately following it, the entire calibration must be reanalyzed, as there is no way of determining what affect the sample matrix would have on any subsequent ICV analysis.

An instrument blank should not precede each CCV – if it's necessary, the instrument rinse/bake times must be adjusted instead. Instrument Blanks may be analyzed after high concentration samples regardless of whether a sample or CCV follows (see analysis/sequence section)

Calibration Verification Acceptance Limits: These standards expire after 24 hours.

		Spike Level (ug/L)	Lower Limit (ug/L)	Upper Limit (ug/L)
ICV	(±10%)	5.00	4.50	5.50
CCV1	(±20%)	2.50	2.00	3.00

I. PREPARE THE SAMPLES & BATCH QC

1) Turn on the digestion block and allow it to come to temperature (95°C).

2) Check samples out of the cold room and allow them to come to room temperature.

3) Calibrate the autopipette at each of the volumes needed to spike the batch QC samples and create the initial calibration standards. Document this calibration in the pipette benchbook.

4) Verify that the balance has been calibrated for the day; if it has not, calibrate it and document the calibration in the balance benchbook.

5) Select sufficient digestion tubes for all of the samples in the batch plus a method blank, blank spike, blank spike duplicate, and two matrix QC samples. Label each tube with a sample number or QC-type.

6) Homogenize a 5-10g subsample and weigh triplicate 0.2g portions of the homogenized subsample into the bottom of the tared digestion tube labeled with that sample number.

7) Record the sample number, container *letter*, and sample weight (to 0.01g), in the benchbook. Place the digestion tube in the rack.

8) For the method blank (MB), simply label an empty digestion tube.

9) Laboratory Control Samples (LCS = Blank Spike) should be spiked between the low and middle level of the calibration standards. Prepare the blank spike (BS) and blank spike duplicate (BSD) by adding 1.25 mL of the 0.100ug/L Hg working standard to each of two empty digestion tubes.

10) Review the job sheets to determine if any of the clients in the batch requested that matrix QC be done on their sample. If so, use that sample for the MS/ MSD, otherwise choose a sample for batch QC so that matrix QC is rotated throughout the laboratory's clients and so that no one client's samples predominate over a period of time.

11) In the absence of alternative specifications, choose samples for matrix spiking such that QC is rotated through clients and projects so that no one client's samples predominate over a period of time. Job/Project instructions are higher priority; if project specific QC specifies selection of specific client samples for MS/MSD, follow this guidance.

12) Prepare the matrix spike (MS) by adding 1.25 mL of the 0.100 ug/mL Hg working standard to a second 0.6 g aliquot of the sample being used for QC.

13) Prepare the matrix spike duplicate (MSD) by adding 1.25 mL of the 0.100 ug/mL Hg working standard to a third 0.6 g aliquot of the sample being used for QC.

14) To each sample and batch QC sample, add 5 mL DI water and swirl to mix.

15) To each sample and batch QC sample, add 5 mL of concentrated aqua regia and swirl to mix. Document the C&T ID of the aqua regia in the digestion benchbook.

16) Place the rack containing the samples, batch QC, and calibration standards in the 95°C digestion block for 2 minutes then remove from the block and cool to about room temperature.

17) Add 25 mL deionized water to each tube. Swirl to mix.

18) Add 7.5 mL of 5% $KMnO_4$ solution to each tube. Mix thoroughly.

19) Put the tubes in the digestion block. Set the block to maintain a temperature of 90-95°C for 30 minutes. In the digestion log, record the time and temperature when the samples are placed into the digestion block and then both again when they are removed from the block.

    Note: The temperature readout of the digestion block will typically be higher than the actual temperature of the digestion tubes, so the actual digestion temperature should be monitored using a thermometer in a reagent blank. The temperature should be within $\pm$ 5°C of the expected temperature. Adjust as necessary until this requirement is met.

20) After about 5 minutes, verify that the digests are still purple in color. If they are not, slowly add just enough granular $KMnO_4$ to make the color persist. Use the same $KMnO_4$ as used to make the liquid solution.

    Note: C&T uses granular $KMnO_4$ because samples high in organics often require too much additional permanganate to make addition of the liquid solution feasible.

21) Cool to about room temperature then add 3 mL of sodium chloride-hydroxylamine-HCL to reduce the excess permanganate.

22) Add 20 mL deionized water, cap and invert to mix.

23) Filter each sample, including batch QC samples, through a clean 0.45µm syringe filter and place the filtrate into a labeled autosampler tube.

24) Document the LIMS standard number of the spikes and all C&T reagent ID's of the reagent in the digestion benchbook.

    Enter the prep data into LIMS and have the entry peer-reviewed before beginning sample analysis so that LIMS can apply the correct prep dilution factor. If any corrections are made to the prep entry after samples are analyzed, that sample data must be reprocessed through LIMS. The digestion benchbook entries should include:

    - C&T sample ID's and unique container identifier,
    - date of sample digestion, initial volume or weight of sample, and final digestate volume,
    - identity of QC samples (spikes, duplicates & LCS),
    - amount of spikes added and LIMS identification numbers of all spiking solutions,
    - a list of all reagents used (C&T ID or manufacturer and lot number),
    - indication of whether or not the digests were filtered after digestion,
    - any unusual occurrences observed during the digestion procedure

25) Use the scanner to scan in the prep information from the digest log and save it under the batch number.

J. ANALYSIS

26) While the samples are digesting, refill the stannous chloride reservoir and make sure the inlet tube is submerged in the reagent; document the reagent ID in the digestion log. Turn on the lamp and allow the instrument to equilibrate for about an hour.

27) Place the filtered samples in an autosampler rack beginning with the initial calibration standards. The initial calibration standards should be analyzed in order of increasing concentration, with the lowest standard at or below the reporting limit and the highest standard defining the upper end of the quantitation range. The correlation coefficient must be $\geq$ 0.995 before the analysis can proceed.

28) CCV standards must be run after every ten samples, or more frequently if desired, and at the end of the sequence; alternate the CCV concentration across the calibration range over the course of the sequence, excluding the high and low ICAL points. A typical sequence is outlined below:

Initial Calibration Standards (run lowest to highest)
Initial Calibration Verification (ICV, second source) standard
Initial Calibration Blank (ICB)
Prep Blank
Blank Spike
Blank Spike Duplicate
Sample
Sample Duplicate
Sample Spike
4 more samples
Continuing Calibration Verification (CCV) standard
Continuing Calibration Blank (CCB)
10 samples
CCV
CCB
etc……

29) Use the data acquisition software to control the autosampler. The instrument will automatically add the 10% stannous chloride reagent and analyze the samples.

More detailed information regarding this procedure may be obtained by consulting the operations manual for the instrument.

30) If a **Serial Dilution (SER)** is needed, measure 2.0 mL of the MSS (unspiked aliquot of the "matrix spike sample") into a digestion tube. Add 8.0mL of the 5% blank (to match the reagent concentrations) and swirl to mix. Decant into an autosampler tube and add to the sequence.

31) If a **Post-Digestion Spike (PDS)** is needed, pipette 10.0 mL of the MSS into a digestion tube. Add 0.5 mL of a 0.1 mg/L standard. Cap and swirl to mix. Decant into an autosampler tube and add to the sequence.

K. Determining Sample Concentrations

Quantitation is based on comparison of the measured absorbance for the sample against the initial calibration curve, with adjustments for the sample preparation concentration factor and instrument dilution factor. See Appendix_4 for example calculations. Concentrations are expressed as micrograms per liter (µg/L) or milligrams per kilogram (mg/kg).

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

1.) **Evaluate the CCV's & CCB's**
Target elements must not be detected in the calibration blanks at any level greater than the method detection limit (DoD QSM specifications allow a wider CCB limit: no analytes present at 2x the MDL). The concentration of the CCV must be varied within the calibration range, over the course of the sequence. If the %D for any element is outside this acceptance window, LIMS will use the following to determine if the associated results are reportable:

- If the CCV fails the %D criterion due to a high response but mercury was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

- If the CCV fails the %D criterion due to a high response and mercury *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.

- If the CCV fails the %D criterion due to a low response, all of the associated samples must be reanalyzed as the low bias may result in false negatives or misquantitation.

2.) **Evaluate the Batch QC Results**

- **Method Blank:** The results for the method (preparation) blank should be < 1/2 RL. If reanalysis confirms the contamination, use the following steps to determine if the sample results may be reported:

  a. If the concentration in the blank is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

  b. If mercury was found in the blank but was not detected in the associated samples, the data may be reported and the problem narrated.

  c. If mercury was found the method blank and was also detected in the associated samples, but the level in the samples is greater than 10x the level in the blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

  d. If mercury was detected in the blank were also detected in the associated samples, but at levels less than 10x the level in the blank, and reanalysis confirms the problem, the samples containing the contaminant must be redigested and

reanalyzed. Initiate a Corrective Action Report (CAR) and redigest and reanalyze all of the affected samples.

- **Blank Spike (BS) and Blank Spike Duplicate (BSD):** The recoveries and RPD should fall within C&T in-house limits, or the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine if the sample results may be reported:

  a. If a high recovery is observed but mercury was not detected in the associated samples, note the failure on the Data Review Checklist and report the data without re-digestion, as the potential high bias does not affect the sample results.

  b. If a high recovery is observed and the samples contain mercury at levels above the reporting limit, those samples must be re-digested.

  c. If a high RPD is observed but the recoveries are within acceptance limits and the samples do not contain mercury, note the failure on the Data Review Checklist and report the data without re-digestion, as the lack of good precision data does not affect ND samples.

  d. If a high RPD is observed and the samples contain mercury at levels above the reporting limits, those samples must be re-extracted.

  Note: Project-specific limits may be different than C&T acceptance limits. For samples submitted for that project or client, the project-specific limits supersede C&T limits.

- **Matrix QC (SSPIKE/SDUP or MS/MSD):** The recoveries and RPD should fall within C&T in-house limits, or the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine any necessary corrective action:

  a. If the concentration in the sample is greater than the linear range and the sample needs to be rerun for quantitation, report the MS/MSD with a LIMS-flag of “>LR” on those recoveries, without reanalysis.

  b. If the concentration in the sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a “>LR” flag to those recoveries. Report the data without reanalysis.

  c. If the concentration is greater than 4x the spiking level, LIMS will apply a “NM” (for “Not Meaningful”) flag to those recoveries. Report the data without reanalysis.

     Note: If the concentration is greater than the spiking level, LIMS will flag and footnote that concentration for the client’s attention.

d. If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

e. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

3.) **Evaluate the Sample Results**

Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

Examine the sample results to verify that all results are within the linear range. If the concentration is greater than the highest calibration standard for that element, use volumetric pipettes to prepare a dilution of the digestate so that the concentration falls in the fall in the middle of the ICAL curve to reduce likelihood of bias found at either end of curve. See appendix 3 below for procedures used to prepare sample dilutions.

If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

4.) **Reviewing & Reporting Results**

1.) Open a web browser and go to the main METALS page.

2.) Under Recent Sequences, choose the instrument and then choose the sequence you want to review.

3.) Check the sequence for any errors. LIMS takes the run information directly from what is typed in the sequence. Typing errors can affect how the data is processed by LIMS.

If you need to make any changes to the sequence, click FIX. Make any needed changes and click UPDATE.

4.) Once everything is correctly processed in the sequence, review the sequence to see that all samples, STDs, and QC meet acceptance criteria. Samples and QC should have an “OK” flag in the QC column of the sequence, otherwise reanalysis may be required. Once the sequence has been reviewed, it must be signed in the REVIEW APP.

5.) Open REVIEW APP for the sequence by choosing the sequence (e.g. MET14 / 05/08/07) under the Review pull down list on the top right corner of the sequence page.

6.) Two windows should open. One window shows the main Review App page. One window shows the documents (e.g. reports, scanned documents, Form 1s, etc).

IMPORTANT! Make sure you are logged in with your own user initials. The current user's initials are displayed on the bottom left corner of the review app window. To logout, click the user initials and Review App will ask you to login.

7.) The sequence is listed on the left frame of the Review App page. Clicking on an item on that list will display the LIMS report and any associated documents.

8.) The analyst adds a comment that the sequence has been verified/reviewed for either all files in the sequence or those files the analyst has reviewed if the sequence is still running.

9.) From another open web browser go back to the main METALS page and click on run chooser.

10.) Enter the login number, analysis or rgroup (metals), and matrix for the job to be reported. Choose the result(s) to be reported for each sample and QC for the job.

11.) Select reports from the top left side of the screen. From the views section in the upper right select possible. This will list the possible reports that can be printed. Select the reports to be printed and print to nowhere. This will select and lock the final results in the LIMS database.

12.) From the REVIEW APP page you left open previously, enter the batch number in the box in front of the search button and click the button.

13.) Review the results and sign the main page of the batch.

14.) From a different open web browser, go back to the main METALS page and enter the job number you wish to report in the search box and select search. Open REVIEW APP for the job by choosing the job (e.g. 214373 METALS Filtrate) under the Review pull down list on the top right corner of the page.

15.) Review the results and sign the main page for the job.

16.) Inform the group leader that the job is ready for review.

17.) When the reviewer or group leader does the final review of the job, the level III or IV packages are generated at that time.

L. POLLUTION PREVENTION

Vent the mercury analyzers into the fume hood. Purchase only sufficient standard and reagent volume to use within the expiration date, to reduce the volume of laboratory waste.

M. WASTE DISPOSAL

Soil samples must be returned to the coldroom for later inclusion in the lab's soil waste stream. Excess digest volume should be discarded in the "Corrosives" waste stream. Excess standard volume should be included in the "Mercury" waste stream.

N. REVISION HISTORY

This is revision 17, Revision 16 was reviewed and changed in the following manner:

- Triplicate 0.2g sampling instead of one 0.5g sample.
- Changed spike level
- Removed alternating CCV levels

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APPENDIX_1: MERCURY SOIL DIGESTION SUMMARY

Sample Weight:	0.6g (± 0.05 g)
Spike:	1.25 mL of HG 0.1 STD
1st Digestion:	+ 5 mL DI water + 5 mL aqua regia 95 °C 2 minutes
2nd Digestion:	+ 25 mL DI water 7.5 mL of 5% $KMnO_4$ solution 90 - 95 °C 30 minutes (add $KMnO_4$ crystals if color fades)
Final:	+ 3.0 mL NaCl-Hydroxylamine HCl repeat until appearance is stable & colorless +20 mL DI water
Filtration:	0.45 µm Syringe Filter, including all batch QC

APPENDIX _ 2: REAGENTS & STANDARDS

The standards and reagents listed below are those in use at the time this procedure was written. Alternative Reagents and Supplies may be substituted provided the substitution is equivalent and meets all calibration, quality control, and traceability requirements.

REAGENTS

Document the preparation of all reagents, including dilutions into DI water, in the reagent prep benchbook; include the date and analyst initials, the name of the reagent, and the manufacturer and lot# in the benchbook entry. Assign a unique ID, based on the type of reagent and the date prepared; record this ID in the digestion benchbook each time the reagent is used. Label each reagent bottle with the date received, date opened, and the expiration date.

Deionized Water, ASTM Type II (ASTM D1193)

Hydroxylamine Hydrochloride ($H_2NOH.HCl$), reagent grade
Fisher Scientific catalog # H330-500
Store in a tightly sealed plastic bottle at room temperature for up to 5 years

Nitric Acid (HNO_3), concentrated, InstraAnalyzed grade
JT Baker catalog # 7697-37-2
Store in a Corrosives cabinet for up to 2 years

Hydrochloric Acid (HCl), concentrated, InstraAnalyzed grade
JT Baker catalog # 7647-01-0
Store unopened bottles in the corrosives cabinet.
Store open bottles under the fume hood for up to five years.

Potassium permanganate ($KMnO_4$), reagent grade, mercury free
Fisher Scientific catalog # P279-212
Store in an amber glass bottle at room temperature for up to 5 years

Potassium persulfate ($K_2S_2O_8$), reagent grade
Fisher Scientific catalog # P281-500
Store at room temperature for up to 5 years

Sodium Chloride (NaCl), reagent grade
Fisher Scientific catalog # S671-500
Store in a tightly sealed plastic bottle at room temperature for up to 5 years

Sulfuric Acid Concentrated (H_2SO_4), concentrated, InstraAnalyzed grade
JT Baker, VWR catalog # JT9691-3
Store in a Corrosives cabinet for up to 2 years

Aqua Regia: *prepare daily*

Prepare immediately before use in a glass bottle

1.) Measure 96 mL concentrated nitric acid (HNO_3) into a glass bottle
2.) Add 32 mL concentrated hydrochloric acid (HCl).
3.) Swirl to mix.

5% Nitric Acid (HNO_3)

1.) Add 50mL concentrated HNO_3 to 950mL deionized water.
2.) Swirl to mix.
3.) Store at room temperature for up to 1 year

5% Potassium permanganate ($KMnO_4$, saturated) by weight: *prepare monthly*

1.) Dissolve 50 g of potassium permanganate in about 400mL DI water in a 1L volumetric flask.
2.) Agitate to dissolve the solids.
3.) Bring to 1,000 mL with DI water.
4.) Cap and invert at least 3 times to homogenize.
5.) Store in an amber glass bottle at room temperature, under a fume hood, for up to one month.

5% Potassium persulfate ($K_2S_2O_8$), by weight: *prepare monthly*

1.) Dissolve 50 g of potassium persulfate in 1000 mL DI water.
2.) Store in an amber glass bottle at room temperature in the hood for up to one month.

Sodium chloride-hydoxylamine hydrochloride solution: *prepare monthly*

1.) Add 120g of sodium chloride to about 200mL of DI water in a 1L volumetric flask.
2.) Add 120g of hydroxylamine hydrochloride.
3.) Agitate to dissolve the solids.
4.) Dilute to 1,000 mL with DI water.
5.) Cap & invert at least 3 times to homogenize.
6.) Store in a glass bottle at room temperature, under a fume hood, for up to one month.

10% Stannous Chloride ($SnCl_2$) “flow reagent” solution: *expires after 5 days*

1.) Add 40g stannous chloride to 200 mL of DI water.
2.) Add 30 mL HCl and swirl to mix.
3.) Add 170 mL of DI water to bring the total volume to 400 mL.
4.) Agitate the mixture to dissolve any residual tin chloride crystals.
5.) Store under a hood, in an amber glass bottle at room temperature for up to 5 days; prepare fresh solution more frequently if it turns slightly yellowish.

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SOURCE STANDARDS

Source standards are those purchased from a chemical manufacturer or vendor and they should be traceable to NIST. For source standards, the LIMS S-name is unique to both the composition (compound list) of the standard *and* to the vendor of that standard. A new S-name must be assigned whenever the composition is changed or when the standard is obtained from a different vendor; the information must then be entered in the "Standard Definitions" table before the new standard is assigned an S#.

Certificates of Analysis should be obtained from the vendor of each source standard; each standard should be traceable to NIST. If the supplier did not provide a certificate, call and request that a copy be faxed. Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is one year from date received.

Enter the lot#, date received, and expiration date of each source standard into LIMS immediately upon receipt, using the Standards Menu "Standard Inventory".

Label the Certificate of Analysis with the LIMS S#, date received and expiration date (if not already listed on vial) then file the certificates the 3-ring binder. Label each vial with the contents, LIMS S#, and expiration date. Store source standards in the vendor's bottle at room temperature, until the expiration date.

Analytes	Concentration	Supplier & Catalog#	LIMS S- Name
Mercury, primary source std. Plasma-Pure Part # 603-8062	1,000 ug/mL	Leeman # 7439-97-6	HG LEEMAN
Mercury, second source std.	1,000 ug/mL	CPI # S4400-1000331	HG SS

WORKING STANDARDS

Working standards are those prepared at C&T. For working standards, the LIMS S-name is not unique to the source standard vendor but *is* unique to the compound list and concentrations contained in the working standard; if the concentration or compounds in the working standard changes, a new S-name, compound list and concentrations must be entered in the "Standard Definitions" table before the standard is logged in and assigned an S#. It is very important to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

Verify that the LIMS expiration date does not exceed the Source Standard expiration date. If it does, change the working standard expiration date to that of the source standard. **The expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**.

In the Standards Benchbook, enter the prep date, LIMS S#, concentration, and volume of each source standard used, the LIMS S-name, final volume and concentration of the working standard, expiration date, and prep chemist's initials.

In LIMS, enter the prep chemist's initials, prep date, and S# of all source standards used to make the working standard; LIMS will then assign a working standard number (S#).

Prepare working standards by diluting source standards to volume in a Class-A volumetric flask. Label the standard vial with the contents, the LIMS WS#, the expiration date of the standard, and the prep chemist's initials.

Procedure:

1.) Prepare all working standards in a **200 mL Class-A volumetric flask**, initially adding about 200mL DI water.
2.) Add 10mL concentrated nitric acid to about 100mL DI water (so the final acid concentration in 200mL final volume will be 5%). Swirl to mix.
3.) Add the volumes and standards listed below to make each standard.
4.) Bring to volume with DI water.
5.) Cap and invert three times to mix. Allow sufficient time, during each inversion, to mix thoroughly.

All volumes given below may be adjusted proportionally to yield more or less of the solution required. All spiking standards should be analyzed to verify their concentration **PRIOR TO USE**. The data from these screens should be stored in the appropriate binder.

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MERCURY STANDARDS PREP

Working Standard & Conc.	WS Conc. (ug/mL)	Using "Source" Std	Add Vol (mL) Source Std	LIMS S-Name	Shelf Life
Primary Source Intermediate	10.0	HG LEEMAN	2.0	HG 10 STD	3 mo
Primary Source Working Std	0.100	HG 10 STD	2.0	HG 0.1 STD	48 hr
2nd Source Intermediate	10.0	HG SS	2.0	HG 10 REF	3 mo
2nd Source Working Std.	0.100	HG 10 REF	2.0	HG 0.1 REF	48 hr

DAILY CALIBRATION STANDARDS

CAL Standard & Conc. (µg/mL)	Final Volume (mL)	Using Source Std	Add Vol (mL) Source Std	LIMS S-Name
CAL-Zero 0.0 ug/L	50	DI water	0.00	
CAL-1 0.2 ug/L	50	HG 0.1 STD	0.10	
CAL-2 0.5 ug/L	50	HG 0.1 STD	0.25	
CAL-3 2.0 ug/L	50	HG 0.1 STD	1.00	
CAL-4 5.0 ug/L	50	HG 0.1 STD	2.50	
CAL-5 10.0 ug/L	50	HG 0.1 STD	5.00	
ICV 5.0 ug/L	50	HG 0.1 REF	2.50	HG ICV
CCV-1 0.5 ug/L	50	HG 0.1 STD or REF	0.25	CCV1 HG
CCV-1 2.0 ug/L	50	HG 0.1 STD or REF	1.00	CCV2 HG
CCV-1 5.0 ug/L	50	HG 0.1 STD of REF	2.50	CCV3 HG

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APPENDIX _ 3: SAMPLE DILUTIONS

If the sample concentration is greater than the highest point in the calibration curve, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range. This dilution must be injected onto the instrument within 24 hours of completion of the digestion; if the digestion was completed more than 24 hours previously, redigest the sample in a new batch and dilute the digestate prior to analysis.

Examine the original sample data to determine what dilution factor will be required to bring the absorbance down to the middle of the calibration range.

Using calibrated auto-pipettes, add the following volumes sample followed by HNO_3 acidified (5% vol:vol) deionized water to a 15mL disposable centrifuge tube. Cap and invert three times to mix, allowing sufficient time for complete mixing with each inversion.

DILUTIONS

Instrument Dilution Factor (IDF)	Digestate Volume	Add Volume (mL) Acidified DI Water	Final Volume (mL)
2	5.0 mL	5.0	10
5	2.0 mL	8.0	10
10	1.0 mL	9.0	10
20	0.50 mL	9.5	10
50	0.20 mL	9.8	10
100	0.10 mL	9.9	10

If a sample should need a dilution of more than 100x, prepare a 100x dilution first, then use that to make subsequent dilutions.

SERIAL DILUTIONS

Instrument Dilution Factor	Using Primary Dilution	Volume of Primary (100x) Dilution	Add Volume (mL) Acidified DI Water	Final Volume (mL)
500	100x	2.0 mL	8.0	10
1,000	100x	1.0 mL	9.0	10
2,000	100x	0.5 mL	9.5	10
5,000	100x	0.2 mL	9.8	10
10,000	100x	0.1 mL	9.9	10

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APPENDIX _4: CALCULATIONS

SAMPLE CONCENTRATION

Raw Amt (ug/L) = (m * intensity) + b

Where: m = first-order coefficient (slope) from initial calibration (LIMS a1)
b = intercept (LIMS a0)

LIMS then applies the sample prep information to calculate the mercury concentration in the sample by dividing the total ug by the prep dilution factor.

Conc (ug/L or mg/Kg) = Raw Amt * Vf / Si * IDF

Where: Vf = Final Digestate Volume (mL)
Si = Sample Initial Volume (mL) or Weight (g)
IDF = Instrument Dilution Factor

Moisture Corrected Results:
Dry Weight Concentration (mg/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

BATCH QC CALCULATIONS

Percent Recovery (%R):
The recovery is the measured concentration divided by the true concentration of the spike.

%Recovery = (Cf – Cs) / (Cws * Vws/ S) *100

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard
S = Sample weight or volume

Relative Percent Difference (RPD):
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

%RPD = |(Cs - Cdup)| / ((Cs + Cdup)/2) * 100

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

For soil MS/MSD's where the sample weights are not weight-targetted, the expected concentations will vary with sample weight (because the same volume of spike standard is being added to different weights of sample) and must be accounted for when calculating RPD:

$$\%RPD = |((Wms/Wmsd)*Cms - Cmsd)| / (((Wms/Wmsd) * Cms + Cmsd)/2) * 100$$

CALIBRATION EQUATIONS

%Drift used calibration verification
Used for calibration verification standards, to determine if the instrument is still in control:

$$\% \text{ Difference (\% Drift)} = \%D = ((C1 - C2) / C1) * 100$$

Where: C1 = Concentration of the Calibration Verification Standard
C2 = Measured concentration

Linear Regression Equations

$$Y = mx + b$$

Where: y = response (Ax for external standard, or Ax/Ais for internal standard)
Where Ax = Area of compound
Ax / Ais = Area of compound divided by area of internal std
x = concentration (Cx for external standard, or Cx/Cis for internal standard)
m = slope
b = intercept

$$\text{Slope (m)} = [(\sum wx_iy_i * \sum w) – (\sum wx_i * \sum wy_i)] / [(\sum w * \sum wx_i^2) – (\sum wx_i * \sum wx_i)]$$

$$\text{Intercept (b)} = y_{avg} – (m * x_{avg})$$

Correlation Coefficient (r)

$$r = \frac{[(\sum w * \sum wx_iy_i) – (\sum wx_i * \sum wy_i)]}{\sqrt{\{[(\sum w * \sum wx_i^2) – (\sum wx * \sum wx_i)] * [(\sum w * \sum wy_i^2) – (\sum wy_i * \sum wy_i)]\}}}$$

$$\text{Coefficient of Determination } (r^2) = r * r$$

Where: x_i = individual values for the independent variable (concentration)
y_i = individual values for the dependent variable (response, area)
w = weighting factor (for no weighting w = 1)
x_{avg} = average of the x-values
y_{avg} = average of the x-values

STANDARDS PREP & OTHER QC

Spiking Batch QC for Waters
Calculate the volume of working standard to add to make the LCS/ MS/ MSD using the following:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where: Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/L)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Volume of Sample (mL)

Spiking Batch QC for Soil
Calculate the volume of working standard to add to make the LCS/ MS/ MSD using the following:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where: Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/Kg)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Mass of Sample (g)

Make a Working Standard from a Source (Stock) Standard:
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where: Vss = Volume of Source Standard (mL) needed to make new standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

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APPENDIX_5: INSTRUMENT CONDITIONS

Instrument:	MET-34, Leeman Hydra AA II MET-14, Leeman Hydra AA	
Argon	Purity	99.99%
	Max Pressure	75 psi
	Flow Rate	0.6 L/min
Oven Radiator Temperature:		125 °C (to maintain 50°C gas temperature)
Lamp Current		6 – 10 mAmps
Auto Sampler		30s sip duration 75s rinse time
Peristaltic Pump Rate		6.0 mL/min Sample 0.8 mL/min Reagent (Stannous Chloride)
Auto-Integration		4 replicates
Baseline Correction		1 point

Note: The conditions listed above were those in use at the time this document was written and may be modified at the analyst's discretion, so long as those changes are documented in the instrument maintenance log.

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APPENDIX_6: INSTRUMENT MAINTENANCE

All parts & supplies can be ordered from LEEMAN; see instrument manual for part numbers. Alternate suppliers and part numbers may be used at the analyst's discretion.

Document all maintenance events must be documented in the instrument maintenance benchbook. Include each of the following in the benchbook:

- reason for the maintenance,
- the initials of the person performing the maintenance and the date performed,
- description of maintenance performed,
- resolution (If the maintenance was due to poor instrument performance, did the maintenance solve the problem?)

Pump windings must be replaced whenever cracked or leaking.

The **drying tube** (gas-liquid separator) should be cleaned by sonicating in 1:1 nitric acid/ DI water for 30 minutes whenever the glass begins to turn yellow from tin chloride precipitation.

The **gas-liquid separator** should be cleaned with spectrophotometric grade isopropyl alcohol if the cell appears smudged or dirty.

The **lamp** should be ordered when the voltage is at 10mV and replaced when the voltage reaches 15mV.

DIGESTION BLOCK INSTRUCTIONS _____

1) Turn on the block.
2) On the keypad, press ENTER/ START.
3) Select AUTO.
4) Set the temperature to 95°C.
5) Allow block to warm up for 20 - 30 minutes.
6) Use a thermometer, in a water-filled digestion tube, to verify that the temperature is 95°C.
7) Adjust the temperature setting as needed so that the digestate temperature will be maintained at 95°C.
8) Place the digestion tubes in the bath.
9) Set the timer for 30 minutes for soil samples (2 hours for water).
10) Document the time and temperature in the digestion log.

APPENDIX_7: USING THE LEEMAN SOFTWARE

Install # 62541
800-533-6267 David Sherwin, Field Service Engineer

The Hg analyzer provides a windows interface that interacts with LIMS once a batch has been entered, prepped and assigned a batch number. Before using the windows interface you must have a LIMS batch number and LIMS prep information.

1. Open the WinHg Runner Program.

   1.1. Click on the icon in the quick launch bar
   1.2. Open the program from Start->Programs->WinHg->WinHg
   1.3. Execute the program file located at C:\Hg\WinHg.exe
   1.4. If all else fails, you can click Start->Run and on command line type "C:\Hg\WinHg.exe"

2. Use the functions under the **Control** tab to control the parts of the instruments such as the pump, gas and lamp. When starting up, go to this tab first. Here you will:

   2.1. Turn on the lamp
   2.2. Turn on the pump (5mL/min)
   2.3. Turn on the gas (0.6 L/min)

   Note: Should you ever get very negative calibrations (~ -2000 or more), use the lamp adjust feature on this tab.

3. Create a new dataset (sequence) by selecting "New Dataset" from File menu.

   3.1. A window will pop up asking for new dataset name.
   3.2. Enter the date in MMDDYY format, e.g. 041305.
   Note: For the second batch to be run on any given day, you will need to append a letter to the name, for example, 041305a, 041305b and so on.
   3.3. Another window will pop-up asking for batch number.
   3.4. Enter your current batch number here.

4. Use the **Standards** Tab to set up the calibration once your standards are made and in the autosampler tray.

   4.1. On left hand side, depress the first six buttons (S1-S6), corresponding to your standards (0.0, 0.2, 0.5, 2.0, 5.0, 10.0),
   4.2. Depress the "Rep 1" button, and click "Stnd Auto".
   4.3. This will begin to establish your calibration curve.
   4.4. Click on the Database (DB) button on the top tool bar.
   4.5. Select the "Cal Curve" Tab to view your calibration curve in progress.

4.6. If your calibration curve has a Rho factor (correlation coefficient) of ≥0.995, click on the "Accept" button on the "Cal Curve" tab of the DB application.

4.7. Print the calibration curve by either:
   a. clicking the print button from the top toolbar of DB application, or
   b. selecting Print from the File menu

4.8. Delete any calibration points not included in the ACCEPTED curve, by selecting the line and clicking on the delete button of the Report tab. *Note:* You can also uncheck the box to the left of the sample ID; however, this change is temporary, as the box will automatically be re-checked as additional samples are run and the list is "refreshed"

5. Use the **Rack Editor** interface to enter information about the samples in the autosampler rack.

6. Use the **Reports** to review the sequence and select the records to submit to LIMS.

6.1. Carefully review the samples in the sequence (or "Record List" as it is referred to on the Report tab of the DB application). Be sure that only the calibration points used in the accepted curve are included in this list. These will be the six StdXRepY that precede the ICV. *Note:* Whenever you reset calibration data (from the standards tab of the Runner application), the curve is reset, but the readings still exist.

6.2. Uncheck any samples you wish to omit.

6.3. Generate the reports by clcking on the "Generate Report" button of the Report Tab.

6.4. Format= Report, Destination = Printer.

6.5. Review this printed report carefully before submitting the data to LIMS. The data that appears on the printout also appears on the **Viewer** Tab of the DB application.

6.6. Format = PRN, Destination= Disk File
Note: The path should be G:\limsinp\met04\batchnumber.PRN.
To set the destination, you must navigate to the .../met04 directory by
   1.5.1 Clicking on the button to the right of the output file path.
   1.5.2 A dialog box will pop-up; in that folder, save the file as its batch number.
   1.5.3 Click Save, or press <Enter>, then Click on "Generate Report".

6.7. After this step is complete, the data is dealt with entirely through the LIMS system.

Note: The default display path is "C:\Hg". This is the local hard-drive directory where the startup.ini, report specifications, protocol parameters and other instrument/system files are housed. Be sure that the "Report Specs" pull down menu displays "DLNEW". This is the format file that enables your insturment to talk to the Laboratory Information Management System (LIMS).

TROUBLE-SHOOTING LEEMAN HYDRA DATA

From the LEEMAN HYDRA AA User's Manual

The WinHG main program has seven tabs that outline its basic functions: main, control, standard, sample, output, report and utility. You will use all of these tabs on a daily basis, except for main and utility.

The typical operation of the HYDRA AA follows a sequence as below:

- Load or create a protocol which defines the operating parameters of the system
- Load or create a dataset where the data will be stored.
- Turn on Gas, Pump, and Hg lamp
- Sample rack editing (Add sample names, Dilution factors, Weight factors, etc.)
- Standard concentration and unit entry.
- Check standard concentration and limits entry.
- Run standards for calibration of HYDRA AA
- Accept calibration curve
- Run samples using calibration stored in protocol.
- Post run reporting of data.

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METALS ANALYSIS BY ICP-MS

EPA 6020 & EPA 200.8

Approved by:

Porter Williams 3/9/12
Department Manager / Date

3/9/12
QA Director / Date

Read & Understood by:

3/9/12
Signature / Date

Signature / Date

3/23/12
Signature / Date

Signature / Date

5.15.12
Signature / Date

Signature / Date

2/12/13
Signature / Date

Signature / Date

Re-Approved by:

2/12/13
Signature / Date

Signature / Date

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METALS by ICP/MS

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This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

METALS ANALYSIS BY ICP-MS
EPA 6020

SCOPE

This document describes the analysis of waters, soils, and hazardous waste for inorganic elements by ICP-MS methods EPA 6020A, EPA 6020 and EPA 200.8. Methods 6020 and 6020A were written by the EPA's Office of Solid Waste with additional guidance for surface water and ground water, as opposed to EPA 200.8 which was written by the EPA's Office of Water specifically for wastewater. EPA 200.8 may also be requested for groundwater samples if the client is planning to discharge the water, with or without additional treatment, into a wastewater stream or into naturally occurring surface waters (bay or river). See Appendix_12 for a summary of EPA 200.8 requirements.

In this analysis, analytes in solution are nebulized and transported to a plasma torch. The ions produced by inductively coupled plasma torch are then measured by mass spectrometry. Background subtraction is used to reduce interferences contributed by the plasma gas, reagents, and sample matrix. No digestion required for dissolved metals. Acid digestion prior to analysis is required for total metals in water, soil, and wastes.

See Appendix_1 for reporting limits and Appendix_8 for a discussion of interferences.

REFERENCES

Analytical Methods:
EPA 6020, Test Methods for Evaluating Solid Waste, SW-846 Update 0, 9/94
EPA 6020A, Test Methods for Evaluating Solid Waste, SW-846 Update 4, Feb 2007
EPA 200.8, Methods for Analysis of Water & Wastewater, EPA 600/4-79-020

Sample Preparation Methods:
EPA 200.8, Methods for Chemical Analysis of Water & Wastewater, EPA 600/4-79-020
EPA 3050B, Acid Digestion of Solid Samples, SW-846 Update 3, Dec.1996
EPA 3010A, Acid digestion of Aqueous Samples and extracts for total Metals for analysis by FLAA or ICP Spectroscopy, rev 1 July 1992

Additional SOP's and Guidance Documents:
DoE Quality Systems Manual, Version 2.2, October 2006
Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Rev 4.2 October 2010

C&T SOP QA 1.6, *Pipet Calibration Check Procedures*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL)*
C&T SOP QA 4.5, *Instrument Detection Limits (IDL) for ICP & ICP-MS*
C&T SOP QA 8.4, *State Program Requirements*
C&T SOP QA 8.5, *DoD Program Requirements*

PRESERVATION & HOLDING TIME

Preservation: HNO_3 to pH $\leq$ 2
Holding Time: 6 months

As of 4/11/07, 40CFR requires sample preservation to be performed within 15 minutes after collection of grab samples, composite samples, or aliquots taken from a composite taken automatically over time. For Dissolved Metals, the samples must be filtered and then preserved within 15 minutes of collection.

If a client requests that the lab filter the samples, a note to this effect should be placed in the case narrative. If the sample pH is >2 upon verification, EPA 200.8 requires that the sample be acidified to pH$\leq$2 *but* the lab must then wait 24 hours before verifying that the acidification was effective by rechecking the pH; if the pH was again >2, the process must be repeated.

SAFETY

Allow digests to cool to room temperature prior to analysis. Sample digests contain concentrated acids and should be handled with caution. Assume all samples, reagents and standards contain hazardous and/ or toxic material and take necessary precautions.

Caution: The torch and interface are hot and should to cool for about 10 minutes before handling.

QC REQUIREMENTS

a.) **Tune Check**
A tune check standard must be analyzed at the beginning of the sequence and every twelve hours to verify that the detector is properly registering the ion masses. See Appendix 6 for details.

b.) **Initial Calibration:**
An initial calibration (ICAL) curve consisting of a calibration blank and at least five standards must be established daily, prior to sample analysis. The lowest concentration standard must be at or below the reporting limit (see Appendix 1 for reporting limits) and the highest standard defines the top of the quantitation range. For samples analyzed by 6020, the correlation coefficient of this curve must be $\geq$ 0.995 and for samples analyzed by 6020A, the correlation coefficient of this curve must be $\geq$ 0.998; if the calibration coefficient criterion is not met, the instrument must be recalibrated.

c.) **Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV):**
An Initial Calibration Verification (ICV) standard obtained from a second supplier must be run at the beginning of each analytical run. A Continuing Calibration Verification (CCV) standard must then be analyzed after every 10 samples, including batch QC samples, and at the end of the sequence.

The concentrations in the ICV and CCV must be within the calibration range and the %D must be < 10% (recoveries between 90-110%) or any samples being quantitated for the failing element must be reanalyzed.

For EPA 6020A analysis: In addition to the midlevel ICV and CCV, a low level ICV should be run following initial calibration. The LLICV and LLCCV should be prepared at the limit of quantification (LOQ) for the calibration with a tolerance of ± 30% of its true value. Both the LLICV and LLCCV should be prepared from the same source as the calibration standards. If any element falls outside these limits, all samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing LLCCV only if the sample result is determined to be less than the specified RL An element may also be reported in the event of a failing LLCCV if the LOQ used for the sample result is greater than or equal to the level of a passing midlevel CCV or a passing LLCCV run at a higher concentration.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the LLCCV must be within ± 20% of its true value.

Note: The USACE recommends that an ICAL standard, or a standard from the same manufacturer as the ICAL standards, be used for the CCVs, to more readily identify problems that are due to changing instrument conditions and are not due to differences between standards.

d.) **Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB):**
An initial calibration blank (ICB) verification must be run at the beginning of each analytical run, with the ICV. A continuing calibration blank (CCB) verification must be analyzed (with each bracketing CCV), after every 10 analytical samples, including batch QC samples, and at the end of the sequence. Target elements should not be detected in the CCB at any level above 3 times the Instrument Detection Limit (IDL).

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the ICB/CCB must be ND > LOD.

e.) **Interference Check Standard A (ICSA):**
An interference check standard (ICS-A) containing only common interferents standard should be analyzed at the beginning of each sequence, after the calibration standards and verifications, to demonstrate that high levels of interferents are not significantly biasing sample results, in either a positive or negative fashion. The determined concentration of the non-interferent should be no more than +RL in either direction. This standard should be repeated every twelve hours and at the end of the sequence.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the measured concentrations of any elements that are not included in the ICS-A standard must be ND > LOD.

f.) **Interference Check Standard A-B (ICSAB):**
An interference check standard (ICS-AB) containing both common interferents and low-level target analytes standard should be analyzed at the beginning of each sequence, after the calibration standards and verifications, to demonstrate that the interference corrections are

effective and are correctly applied. This standard should be repeated every twelve hours and at the end of the sequence to demonstrate that instrument conditions have not significantly changed over the course of the sequence.

The recovery of the spiked elements in this standard must be within 80-120% recovery; see Appendix_6 for additional discussion.

g.) **Batch QC:**
The following quality control (QC) samples must be prepared in the same manner as the analytical samples at a rate of once per twenty or less samples. For EPA 6020, and once per 10 samples or less for method 200.8. C&T in-house acceptance limits are updated semi-annually; based on control charts of the previous year's data. See the associated SOP '6020 QC Limits, Table-1' for the current 6020 limits. For EPA 200.8, the QC recovery limits are specified in the method and can be found in Appendix_12.

Note: Project-specific quality assurance plans may have different criteria. If so, those requirements supersede this SOP for all samples related to that project.

Method Blank (BLANK): The purpose of the method blank is to ensure that the digestion and analysis process does not in any way contaminate the analytical samples. Deionized water is carried through the entire digestion process and analyzed. For EPA 6020, the results for the preparation blank should be <1/2 RL and must be < RL for all target elements. If the sample result for that compound(s) is greater than ten (10) times the amount found in the method blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis. If the sample result for that compound(s) is greater than the reporting limit but less than ten (10) times the amount found in the associated method blank, the samples must be re-digested and reanalyzed. see the 'Quantitative Analysis' section below for further details.

EPA 200.8 Method Note: Method 200.8 requires redigestion and reanalysis of any sample associated with a method blank containing laboratory contamination greater than 2.2x the MDL for that element and <10x the concentration in the sample. If the sample is ND for the blank contaminant or is >10x the level found in the blank, the sample may be reported without corrective action.

Blank Spike (BS) and Blank Spike Duplicate (BSD): The purpose of the blank spikes is to demonstrate that the sample preparation and analysis procedures are accurate (recovery) and precise (RPD) in the absence of matrix interferences. A known concentration of each element is added to deionized water and carried through the entire digestion and analysis process. For EPA 200.8, the recoveries must fall within the method-specified limits, which are listed in Appendix_12. For EPA 6020, the recoveries and RPD should fall within C&T in-house limits. If the acceptance limits are not met, the samples associated with it may need to be redigested and reanalyzed; see the 'Quantitative Analysis' section below for further details.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD): The purpose of the matrix spikes is to demonstrate that the sample preparation and analysis procedures are accurate (recovery) and precise (RPD) in the possible presence of matrix interferences. A known concentration of each element is added to a real-world sample and carried through the entire digestion and analysis process. For EPA 200.8, the recoveries must fall within the method-specified limits, which are listed in Appendix 12. For EPA 6020, the recoveries and RPD should fall within C&T in-house limits. If the acceptance limits are not met, the samples associated with it may need to be redigested and reanalyzed; see the 'Quantitative Analysis' section below for further details.

If the concentration of any element in the spiked sample is greater than four times the spiking level, the recovery is considered 'Not Meaningful' and LIMS will place an "NM" flag on the report. *Note:* For USACE projects, if the concentration of a target compound is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

Note: For Arizona samples, the RPD criterion is < 20% for all matrices.

Sample Duplicate (SDUP): For leachates or other samples known to contain high levels of target analytes, a sample duplicate and sample spike may be analyzed in place of the MS/MSD. The selected sample is prepared and analyzed in duplicate to determine the precision of the sample preparation and analysis process in the presence of potential matrix interferences. If the RPD exceeds C&T RPD limits for a majority of the analytes of interest, the source of the error must be identified and narrated or the affected samples redigested.

Note: For Arizona samples, the RPD criterion is < 20% for all matrices.

Sample Spike (SSPIKE): A second aliquot of a sample is spiked with a known concentration of elements to determine the accuracy of the sample preparation and analytical process in the presence of potential matrix interferences. For EPA 200.8, the recoveries must fall within the method-specified limits, which are listed in Appendix 12. For EPA 6020, the recoveries and RPD should fall within C&T in-house limits. If the acceptance limits are not met, the samples associated with it may need to be redigested and reanalyzed; see the 'Quantitative Analysis' section below for further details.

If the concentration of any element in the spiked sample is greater than four times the spiking level, the recovery is considered 'Not Meaningful' and LIMS will place an "NM" flag on the report. *Note:* For USACE projects, if the concentration of a target compound is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

h.) **Sample Interference Verifications:**
The following matrix interference verifications should be analyzed with each batch of samples or whenever the analyst suspects that sample viscosity, salt content, or other matrix interferences are likely.

Serial Dilutions: Analysis of a 5x dilution should agree within ± 10% (90-110% recovery) of the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference should be suspected.

Post Digest Spikes: An analyte spike added to a portion of a prepared sample digest, or its dilution, should be recovered to within 75-125% of the known value for samples analyzed by 6020 and 80 – 120% for samples analyzed by 6010A. The spike addition should produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits a matrix effect should be suspected and the sample should be diluted (to dilute out the matrix interference); dilution results should agree with the original results within 90-110%.

i.) *Method Modification:* C&T does not use the concept of **Linear Dynamic Range**, in which the upper limit of quantitation is established through extrapolation. Unlike ICP-AES methods (6010 and 200.7), the upper limit of the ICP-MS calibration range is highly dependent on the voltage of the electron multiplier in the detector, which may change any time maintenance is performed or the instrument tuned. For this reason, the upper end of the quantitation range must be established by a daily multi-point calibration curve. The highest calibration standard, which is at a much lower concentration than those used as the "high standard" in an extrapolation scenario, determines the top of the quantitation range for each mass/charge (m/z) ratio utilized, by determining the signal response from four different concentrations across the range.

j.) **Interference equations** must be verified daily and updated whenever the mass axis and resolution are updated or after the detector is cleaned. The Agilent software includes auto-correct programs for the elements listed in the methods. Fine tuning may be performed during the tuning sequence.

k.) **Instrument detection limits (IDL)** must be determined quarterly by analyzing a reagent blank for seven consecutive injections on each of 3 non-consecutive days. The RSD is determined for each element on each day then the 3 RSD's are averaged to determine the Instrument Detection Limit. The IDL must be less than the water MDL for that instrument.

l.) **Method detection limits (MDL) based on 7 replicates of a low-level laboratory control sample that are digested and analyzed annually. See the QA SOP for** Establishing and Validating Method Detection Limits or Limits of Detection for details.

J) **Limit of Detection (LOD)**, must be determined quarterly and is based on a laboratory control sample (MDL verification standard) that is spiked 2 to 4 times the MDL, digested and analyzed on every instrument. See the QA SOP for Establishing and Validating Method Detection Limits or Limits of Detection for details.

k.) **Limit of Quantitation (LOQ)** must be determined quarterly and is based on a laboratory control sample that is spiked 1 to 2 times the reporting limit. It is only analyzed once per method. See the QA SOP for Establishing and Verifying the Limit of Quantitation for details.

EQUIPMENT (see Appendix_9 for instrument conditions):
Agilent 7500 ICP-MS Integrated Sample Introduction System
MET-06: Agilent 7500ce ICP-MS with Octopole Reaction System
MET-16: Agilent 7500ce ICP-MS with Octopole Reaction System
0.45um x 25 mm GD/X Disposable PTFE syringe filter, VWR, Whatman # 28138-164
10 ml disposable syringe, BD syringe, VWR # BD309604

DAILY MAINTENANCE

- Replenish the rinse-water reservoir daily with acidified (1% HCl plus 1% HNO_3) deionized water.
- Verify that the Drain Vessel is empty. If it is not, empty the vessel and rinse it thoroughly with DI water.
- Change the Sample and Internal Standard pump windings daily. Soak Tygon Sample and Internal Standard pump windings in acidified (5%HCl plus 5%HNO_3) deionized water for at least 2 hours prior to use.
- Change the drain pump winding every monthly, or whenever pump winding become flattened or damaged.

DAILY INSTRUMENT SEQUENCE

1.) Turn the plasma on.

2.) Allow at least 30 minutes for the instrument to equilibrate.

3.) Analyze the Tuning Solution. See Appendix_6 for discussion and acceptance criteria.

4.) Using the 50 µg/L calibration standard (MET-06 & MET-16), run the Pulse/ Analog program to establish the concentration (intensity) at which the instrument should switch from Pulse to Analog mode.

Note: The Agilent-7500 operates in two modes, pulse-mode for typical low-level samples and analog-mode for high-level samples. The instrument automatically switches between the two modes based on the voltages encountered during sample analysis; the mode used for quantitation may vary on an analyte-by-analyte basis. The raw instrument data will include a data qualifier flag indicating which mode was used for quantitation, A(nalog), P(ulse), or M(ixed).

5.) Start the sequence by analyzing the 10 µg/L 6020 Tuning Check Standard. The instrument must meet the criteria listed in Appendix_6 before analysis can continue.

6.) If EPA 200.8 samples will be included in the sequence, analyze the 10 µg/L 200.8 Tuning Check Standard. The instrument must meet the criteria listed in Appendix_6 before samples can be analyzed by EPA 200.8.

7.) Calibrate the ICP by running a calibration blank followed by at least five calibration standards, in increasing order of concentration, at levels that bracket the quantitation range; the lowest standard must be at or below the reporting limit and the highest standard determines the upper end of the quantitation range (see Appendix_5 for preparation of these standards).

200.8 Method Modification: Method 200.8 only requires a 3-point calibration however C&T standard practice is to run 5 or more points, to better establish the curve.

8.) Send the ICAL data to LIMS.

9.) Review the ICAL summary to determine if the sequence can be continued:

- For samples analyzed by 6020, the correlation coefficient for each element must be ≥ 0.995.
- For samples analyzed by 6020A, the correlation coefficient for each element must be ≥ 0.998
- The highest concentration standard may be omitted so as long as there are at least three points remaining and the remaining highest point defines the top of the calibration range (any digests which exceed this concentration must be diluted and reanalyzed).
- The lowest concentration standard may be omitted from curve if, *and only if*, the resulting lowest standard is at or below the reporting limit for samples and there are at least three points remaining.
- Mid-point standards may not be omitted simply to improve the correlation coefficient. They may, however, be reanalyzed if poor aspiration is suspected. The reanalysis must occur immediately after the curve so long as no sample digests were analyzed since the last calibration standard and all elements are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular element.

10.) Analyze an Initial Calibration Verification (ICV) standard, obtained from a second manufacturer, at a different concentration than the calibration standard. The result must be within ±10% of the true value (90-110% recovery) or the instrument must be recalibrated.

11.) **For Method 6020A analysis**: Analyze a low level ICV (LLICV) after running the initial calibration. The LLICV should be prepared at the limit of quantification (LOQ) for the calibration with a tolerance of ± 30% of its true value. The LLICV should be prepared from the same source as the calibration standards. If any element falls outside these limits, all

samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing LLICV only if the sample result is determined to be less than the specified RL An element may also be reported in the event of a failing LLICV if the LOQ used for the sample result is greater than or equal to the level of a passing midlevel CCV or a passing LLICV run at a higher concentration.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the LLICV must be within ± 20% of its true value.

12.) Analyze an Initial Calibration Blank (ICB) consisting of deionized water acidified with 1% HCl and 1% HNO_3. All elements must be less than the reporting limit before the instrument is calibrated.

13.) Analyze the ICS-A to demonstrate that high levels of interferents are not biasing low-level quantitation. The determined concentration of the non-interferent should be ≤RL in either direction (+/-), except for those elements that are considered by the manufacturer to be 'trace' contaminants of the high level elements and are listed on the Certificate of Analysis.

If this standard fails acceptance criteria**,** adjust either the mass resolution, or parameters in the Hydrogen or Helium modes.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the measured concentrations of any elements that are not included in the ICS-A standard must be ND > LOD, except for those elements that are considered by the manufacturer to be 'trace' contaminants of the high level elements and are listed on the Certificate of Analysis.

200.8 Method Modification: EPA 200.8 does not discuss use of this type of standard, however C&T follows SW-846 guidance and includes them in every analytical sequence as discussed above.

14.) Analyze the ICS-AB to demonstrate that quantitation in the presence of high-level interferents is acceptably accurate. The determined concentrations of the non-interferent elements in the ICS-AB must be within ± 20% of the true values. If this standard does not meet the acceptance criteria, the analysis must be terminated, the method corrected for the interference, and the analysis restarted from the Tuning Check Standard.

200.8 Method Modification: EPA 200.8 does not discuss use of this type of standard however C&T follows SW-846 guidance and includes them in every analytical sequence as discussed above.

15.) Now actual samples may be run. Collect the digests, job sheets, LIMS prep sheet, and copy of the benchbook page from the prep chemists. Sign the "Received by" line on the LIMS prep sheet to maintain internal chain-of-custody of the digests.

16.) Decant the digests into autosampler tubes labeled with the sample number and dilution factor. Depending on which instrument is being run, digestates may need to be diluted to compensate for salinity.

Water samples are typically analyzed at a 1:10 dilution, and soil samples at a 1:50 dilution; the dilutions should be made with acidified (1% HCl plus 1% HNO_3).

Prepare dilutions as described in Appendix_2. Don't use the auto-dilution feature on the instrument, as results obtained using this feature have shown poor agreement with additional dilutions or undiluted results obtained by optical ICP-AES.

Note: If any of the digests contain suspended particles, which could clog the injection tubing, filter the digests *and* the associated method blank and LCS or BS/BSD through acid-washed syringe filters prior to adding them to the sequence.

17.) Nebulize each sample for 30 seconds or until a steady signal is observed, prior to collecting data, and then collect 3 integrations. The instrument will flush the system for approximately 90 seconds between samples and will go on to the next autosampler tube once the signal has stabilized.

18.) Analyze a continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the sequence.

The CCV result must be within ±10% of the true value (90-110% recovery) or the instrument must be recalibrated and any samples analyzed after the last passing CCV must be reanalyzed.

Target elements should not be detected in the CCB above 3x the IDL; any samples bracketed by a contaminated CCB and being analyzed for a detected element must be reanalyzed.

19.) **For EPA Method 6020A analysis:** Analyze a low level CCV (LLCCV) following the ICV. The LLCCV should be prepared at the limit of quantification (LOQ) for the calibration with a tolerance of ± 30% of its true value. The LLCCV should be prepared from the same source as the calibration standards. If any element falls outside these limits, all samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing LLCCV only if the sample result is determined to be less than the specified RL An element may also be reported in the event of a failing LLCCV if the LOQ used for the sample result is greater than or equal to the level of a passing midlevel CCV or a passing LLCCV run at a higher concentration.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the LLCCV must be within ± 20% of its true value.

20.) Analyze the Tune Check Standard, ICS-A, and ICS-AB at the beginning of every 12-hour shift. The standards must pass the acceptance criteria listed in Appendix_6 or any standards analyzed after the failing standard must be reanalyzed.

21.) Analyze the ICS-A, and ICS-AB at the end of every 12-hour shift. The standards must pass the acceptance criteria listed in Appendix_6 or any samples analyzed after the last passing set of ICS standards must be reanalyzed.

Analytical Sequence

A typical analytical sequence looks like the following:

Tuning Solution
200ppb Pulse/Analog Solution (or 50ppb for MET06)
10ppb Tune Check Standard
Initial Calibration Blank
Initial Calibration Standards (0.25, 0.5, 1, 10, 100, 200 ug/L)
Initial Calibration Verification (ICV)
Initial Calibration Blank (ICB)
Interference Check Standard A (ICSA)
Interference Check Standard A/B (ICSAB)
Method Blank (MB)
Blank Spike (BS)
Blank Spike Duplicate (BSD)
QC Sample (MSS)
Matrix Spike (MS)
Matrix Spike Duplicate (MSD)
Serial Dilution (SER)
.. 3 more samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
.. 10 samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
.. 10 samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
…more samples and CCV's (until 12-hour tune clock runs out)
Tune Check Standard
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
Interference Check Standard A (ICSA)
Interference Check Standard A/B (ICSAB)
.. 10 samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)

… etc.
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
Interference Check Standard A (ICSA)
Interference Check Standard A/B (ICSAB)

The sequence must end with a CCV, CCB, ICS-A, and ICS-AB regardless of the number of samples that have been analyzed.

After the sequence is complete, the Agilent software will automatically turn off the instrument.

LIMs automatically analyzes 2 ICBs and CCBs and always chooses the second run, even when specific elements fail blank criteria, we continue running samples for passing elements. Corrective action is taken when the analyst judges it to be warranted.

QUANTITATIVE ANALYSIS
The Agilent ICP-MS automatically adds internal standard, which helps compensate for viscosity and transport interferences. The sample is then transported through the nebulizer and vaporized in the plasma. The spectrometer measures the mass-to-charge ratio (m/z) of the resulting ions. For all samples and standards, the instrument collects information for 3 integrations and reports the average intensity of these integrations, along with the %RSD between the integrations. The RSD between the integrations must be ≤ 20% or the sample must be reanalyzed. If RSD for the reanalysis is again greater than 20%, report the analysis with the lowest RSD and narrate the problem on the Data Review Checklist; LIMS will apply a qualifier flag to reported result on the final form.

Method Modification Note: Methods 6020, 6020A, and 200.8 do not specify a maximum RSD, so C&T has chosen 20% as the RSD limit to standardize ICP criteria.

Quantitation is based on comparison of the intensity of the target element and internal standard to the initial calibration curve for that element, with adjustments for the sample preparation concentration factor and instrument dilution factor. See Appendix_11 for example calculations. Concentrations are expressed as micrograms per liter (µg/L) or milligrams per kilogram (mg/kg).

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

a.) **Evaluate the Internal Standard Recoveries**
Internal standards are added to the samples and standards at the time of analysis by a second on-line channel of the peristaltic pump. See Appendix_4 for internal standard assignment and comments.

The internal standard recoveries are determined by dividing the intensity observed in the sample by that observed in the first Initial Calibration Standard in the ICAL, which is a zero standard. The internal standard recoveries associated with all required/ reported elements

should be within the recovery limits listed below or the sample should be diluted and reanalyzed.

6020 ISTD Recovery:	30-120%
200.8 ISTD Recovery:	60-125%

The CCV and CCB internal standard recoveries associated with all reported sample results must be within these limits or the instrument must be recalibrated and any samples analyzed after the last passing CCV reanalyzed. Note: If an internal standard fails but is not assigned to any of the requested elements for that sample, the sample does not have to be reanalyzed.

b.) **Evaluate the CCV Results**
The concentration of the CCV must be within the calibration range, with %D $\leq$ 10% (recoveries between 90-110%). If the %D for any element is outside this acceptance window, LIMS will use the following to determine if the associated results are reportable:

- If the failing element is not a target analyte for the associated samples, sample results should be reported without reanalysis.
- If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.
- If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.
- If the compound fails the %D criterion due to a low response, the sample must be reanalyzed as the low bias may result in false negatives or misquantitation.

c.) **Evaluate the CCB Results**
Target elements should not be detected in the calibration blank at any level greater than 3 times the Instrument Detection Limit. If target elements are detected, use the following to determine if the associated results are reportable:

- If the detected element is not a target analyte for the associated samples, sample results should be reported without reanalysis.
- If the detected element was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.
- If the detected element *was* also detected in any of the associated samples at levels above the reporting limit but the sample concentrations were greater than 20 times the concentration in the instrument blank, the samples may be reported.

- If the detected element *was* also detected in any of the associated samples at levels between the reporting limit and 20 times the reporting limit, the samples must be reanalyzed.

d.) **Evaluate the Batch QC Results**

- **Prep Blank:** For 6020, the results for the preparation blank should be <1/2RL and must be <RL for all target elements. For 200.8, the results must be <2.2x the MDL. If reanalysis confirms the contamination, use the following steps to determine if the sample results may be reported:

  a. If the concentration of the contaminant is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

  b. If the target element(s) found in the blank was not detected in the associated samples, the data may be reported and the problem narrated.

  c. If the target element(s) found the method blank was also detected in the associated samples, but the level in the samples is greater than 10x the level in the blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

  d. If the target element(s) detected in the blank were also detected in the associated samples, but at levels less than 10x the level in the blank, and reanalysis confirms the problem, the samples containing the contaminant must be re-batched and reanalyzed. Initiate a Corrective Action Report (CAR) immediately so that re-digestion can begin within the clients requested turn-around time, if necessary.

- **Blank Spike (BS) and Blank Spike Duplicate (BSD):** For 6020, the recoveries and RPD should fall within C&T in-house limits; for 200.8, the recoveries must fall within the method-specified acceptance limits (listed in Appendix 12) and the RPD must be within C&T in-house limits. If these limits are not met, the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine if the sample results may be reported:

  a. If the samples are being analyzed for only a subset of the target element list (ie: lead only, LUFT 5, etc.) and those elements all pass acceptance criteria, the data may be reported without further corrective action.

  b. If a high recovery is observed but that element was not detected in the associated samples, note the failure on the Data Review Checklist and report the data without re-digestion, as the potential high bias does not affect the sample results.

  c. If a high recovery is observed and the samples contain that element at levels above the reporting limits, the samples containing that element must be re-digested.

d. If a high RPD is observed but the recoveries are within acceptance limits and the samples do not contain that element, note the failure on the Data Review Checklist and report the data without re-digestion, as the lack of good precision data does not affect ND samples.

e. If a high RPD is observed and the samples contain that element at levels above the reporting limits, those samples containing that element must be re-extracted.

- **Matrix QC (SSPIKE/SDUP or MS/MSD):** For 6020, the recoveries and RPD should fall within C&T in-house limits; for 200.8, the recoveries must fall within the method-specified acceptance limits (listed in Appendix_12) and the RPD must be within C&T in-house limits. If these limits are not met, the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine any necessary corrective action:

a. If the concentration of a target element in the sample is greater than the linear range and the sample needs to be rerun for just that compound, report the MS/MSD with a LIMS-flag of ">LR" on those recoveries, without reanalysis.

b. If the concentration of a target element in the sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a ">LR" flag to those recoveries. Report the data without reanalysis.

c. If the concentration of a target element is greater than 4x the spiking level, LIMS will apply a "NM" (for "Not Meaningful") flag to those recoveries. Report the data without reanalysis.

Note: If the concentration of a target compound is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

d. If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

e. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

- **Serial Dilutions:** Analysis of a 5x dilution should agree within ± 10% (90-110% recovery) of the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference, such as viscosity of the digestate, should be suspected. If for a given project, all elements repeatedly show an increased response in the Serial Dilution, when compared to the initial run, discuss the problem with the Department Manager and/or the Project Manager for that account, as the samples may require routine dilution to overcome viscosity effects.

- **Post Digest Spikes:** An analyte spike added to a portion of a prepared sample digest, or its dilution, should be recovered to within 75-125% of the known value for samples analyzed by 6020 and 80 – 120% for samples analyzed by 6020A. The spike addition should produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. If the recoveries of all elements shows a uniform bias in either direction, make and analyze a new spike to verify that it was prepared correctly. If the spike is not recovered within the specified limits a matrix effect should be suspected and the sample should be diluted (to dilute out the matrix interference); dilution results should agree with the original results within 90-110%.

e.) **Evaluate the Sample Results**

Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

Examine the sample results to verify that all requested elements are reported on the internal user report and that the results are within the linear range. If the concentration of any requested target element is greater than the highest calibration standard for that element, use volumetric pipettes to prepare a dilution of the digestate so that the highest target element is in the upper half of the calibration range. If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

Examine the sample results to verify that the RSD between exposures is less than 20%. Any sample result with a duplicate exposure RSD greater than 20% must be reanalyzed. If the RSD is still greater than 20%, report the exposure with the lower %RSD. Any sample with requested element concentrations above the linear range must be diluted and reanalyzed.

Method Modification Note: Methods 6020, 6020A, and 200.8 do not specify a maximum RSD, so C&T has chosen 20% as the RSD limit to standardize ICP criteria. The USACE requires that the RSD be ≤10% for concentrations for samples with concentrations equal to or greater than ten times MDL and ≤ 20% for samples with concentrations less than ten times the MDL.

f.) **Assemble the Data Package**

After all samples and necessary dilutions have been analyzed, print the final report forms on “page 2” letter head paper. Review these forms to make sure that the correct results were reported and that there are no elements marked “N/A” (results “not available”).

Verify that the prep sheets are complete and signed, that all necessary sample information is present (see section below for details), then complete and sign the “Data Review Checklist”. Submit the data package to the Department Manager or QC Chemist for second-party review. Any changes made by the second-party reviewer must be individually initialed and dated by the reviewer. The second party reviewer must initial and date each user report, make any additional comments on the case narrative and initial and date the completed checklist.

DOCUMENTATION & PEER REVIEW

The raw ICP-MS data must be labeled with the analysts name, the date analyzed, the instrument units, and the batch ID. The ICP-MS raw data, a copy of the sequence log, LIMS calibration summaries, and copies of the prep logs should be clipped together and filed in the department's data files. A copy of the sequence log must be placed in the bound benchbook.

a.) **Sample Prep Documentation**

A copy of the digestion benchbook page for the sample digestion must be included with the reported data. The digestion benchbook entries should include:

- C&T sample ID's and unique container identifier,
- date of sample digestion, initial volume or weight of sample, and final digestate volume,
- identity of QC samples (spikes, duplicates & LCS),
- amount of spikes added and LIMS identification numbers of all spiking solutions,
- a list of all reagents used (C&T ID or manufacturer and lot number),
- indication of whether or not the digests were filtered after digestion,
- any unusual occurrences observed during the digestion procedure

b.) **"Level 2" Data Package**

A complete data package for a "Level 2" report includes the following:

- C&T Job sheet,
- Final reports for samples and batch QC
- "Inorganics Data Review" checklist,
- Any associated Corrective Action Report.
- Sample LIMS user reports,
- Batch QC LIMS user reports,
- Sequence log clipped to LIMS User Reports for the samples and batch QC from that sequence
- LIMS prep log,
- Digestion log,

c.) **"Level 3" Data Package**

A complete data package for a "Level 3" report includes all of the items listed below, in the following specific order:

- C&T Job sheet,
- Metals "reporting grid" - showing which element was reported from which run,
- Final reports for samples and batch QC
- "Inorganics Data Review" checklist,
- Any associated Corrective Action Report.
- Sample LIMS user reports,
- Batch QC LIMS user reports,
- Serial Dilution LIMS user report,
- Post-Digestion Spike LIMS user report (if necessary),
- Sequence Package, which contains the following, clipped together:

- Sequence summary
- ICAL summary,
- ICV, ICB, CCV, CCB, ICS-A and ICS-AB summaries, in sequential order

- LIMS prep log & copy of digestion benchbook page
- LIMS User Reports for the samples and batch QC

d.) **"Level 4" Data Package**

A complete data package for a "Level 4" report includes all of the items listed below, in the following specific order:

- C&T Job sheet,
- Metals "reporting grid" - showing which element was reported from which run,
- Final reports for samples and batch QC
- "Inorganics Data Review" checklist,
- Any associated Corrective Action Report.
- Sample LIMS user reports,
- Batch QC LIMS user reports,
- Serial Dilution LIMS user report,
- Post-Digestion Spike LIMS user report (if necessary),
- Sequence Package, which contains the following, clipped together:
  - Sequence summary
  - ICAL summary,
  - ICV, ICB, CCV, CCB, ICS-A and ICS-AB summaries, in sequential order
- LIMS prep log & copy of digestion benchbook page
- Sample Data package (includes the following, in the following specific order, clipped together):
  - Sequence log
  - Sample LIMS user reports
  - Serial Dilution LIMS user report
  - Batch OC LIMS user reports (Blank, BS/BSD, MS/MSD)
  - Raw QC and sample data, job specific, in sequential order, excluding flushes and rinses

POLLUTION PREVENTION

Prepare only sufficient standard and reagent volume to use within the shelf-life of the standard to reduce the volume of waste generated by the laboratory.

WASTE DISPOSAL

All digests are kept for at least 6 months prior to disposal. After 6 months, the digests are included in the 'Corrosives' waste stream. Expired standards should also be discarded into the 'Corrosives' waste stream.

REVISION HISTORY

This is rev 9. Rev 8 was changed as follows:

- Removed: LLICV and LLCCV run at end of analytical sequence.

APPENDIX_1: ELEMENTS & STANDARD REPORTING LIMITS

ICP-MS Metals
EPA 6020

CAS #		Element	Reporting Limit*		CAS #		Element	Reporting Limit	
			µg/L	mg/Kg				µg/L	mg/Kg
7429-90-5	Al	Aluminum	50	10	7439-95-4	Mg	Magnesium	50	10
7440-36-0	Sb	Antimony	1	0.25	7439-96-5	Mn	Manganese	1	0.25
7440-38-2	As	Arsenic	1	0.25	7439-98-7	Mo	Molybdenum	1	1.3
7440-39-3	Ba	Barium	1	0.25	7440-02-0	Ni	Nickel	1	0.5
7440-41-7	Be	Beryllium	1	0.25	7440-09-7	K	Potassium	50	25
7440-43-9	Cd	Cadmium	1	0.25	7782-49-2	Se	Selenium	1	0.25
7440-70-2	Ca	Calcium	50	10	7440-22-4	Ag	Silver	1	0.25
7440-47-3	Cr	Chromium	1	0.50	7440-23-5	Na	Sodium	50	25
7440-48-4	Co	Cobalt	1	0.25	7440-28-0	Tl	Thallium	1	0.25
7440-50-8	Cu	Copper	1	0.25	7440-62-2	V	Vanadium	1	0.25
7439-89-6	Fe	Iron	50	10	7440-66-6	Zn	Zinc	1	1.0
7439-92-1	Pb	Lead	1	0.25					

Method Modification: C&T includes the elements listed in 6020 (1994) plus the following elements that were added to the 6020A draft version (1998) that has not yet been promulgated: Ca, Fe, Mg, K, Se, Na, and V.

Low Level ICP-MS Metals
EPA 6020 (by special request)

CAS #		Element	Reporting Limit*		CAS #		Element	Reporting Limit*	
			µg/L	mg/Kg				µg/L	mg/Kg
7429-90-5	Al	Aluminum	50	10	7439-95-4	Mg	Magnesium	50	10
7440-36-0	Sb	Antimony	0.25	0.25	7439-96-5	Mn	Manganese	0.25	0.25
7440-38-2	As	Arsenic	0.5	0.25	7439-98-7	Mo	Molybdenum	0.5	1.3
7440-39-3	Ba	Barium	0.25	0.25	7440-02-0	Ni	Nickel	0.25	0.5
7440-41-7	Be	Beryllium	0.25	0.25	7440-09-7	K	Potassium	50	25
7440-43-9	Cd	Cadmium	0.25	0.25	7782-49-2	Se	Selenium	0.5	0.25
7440-70-2	Ca	Calcium	50	10	7440-22-4	Ag	Silver	0.25	0.25
7440-47-3	Cr	Chromium	0.5	0.50	7440-23-5	Na	Sodium	50	25
7440-48-4	Co	Cobalt	0.25	0.25	7440-28-0	Tl	Thallium	0.5	0.25
7440-50-8	Cu	Copper	0.5	0.25	7440-62-2	V	Vanadium	1	0.25
7439-89-6	Fe	Iron	50	10	7440-66-6	Zn	Zinc	1	1.0
7439-92-1	Pb	Lead	0.25	0.25					

* These lower reporting limits may be analyzed upon special request and may only be used if the current MDL is <1/3 the requested RL and the lowest ICAL is at or below the requested RL.

APPENDIX_2: SAMPLE DILUTIONS

If the sample concentration is greater than the highest point in the calibration curve, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range. Examine the original sample data to determine what dilution factor will be required to bring the absorbance down to the middle of the calibration range.

Using calibrated auto-pipettes, dilute the following volumes of sample digestate in a disposable centrifuge tube; adjust the acid concentration as needed so that the final acid concentration in the diluted aliquot is 1% HNO_3 and 1% HCl. Cap and invert three times to mix, allowing sufficient time for complete mixing with each inversion.

DILUTIONS

Instrument Dilution Factor (IDF)	Digestate Volume	Add Volume (mL) Acidified DI Water	Final Volume (mL)
2	5.0 mL	5.0	10
5	2.0 mL	8.0	10
10	1.0 mL	9.0	10
20	0.50 mL	9.5	10
50	0.20 mL	9.8	10
100	0.10 mL	9.9	10

If a sample should need a dilution of more than 100x, prepare a 100x dilution first, then use that to make subsequent dilutions.

SERIAL DILUTIONS

Instrument Dilution Factor	Using Primary Dilution	Volume of Primary (100x) Dilution	Add Volume (mL) Acidified DI Water	Final Volume (mL)
500	100x	2.0 mL	8.0	10
1,000	100x	1.0 mL	9.0	10
2,000	100x	0.5 mL	9.5	10
5,000	100x	0.2 mL	9.8	10
10,000	100x	0.1 mL	9.9	10

APPENDIX_3: QUANTITATION ISOTOPES

Element	Quantitation Isotope	Secondary Isotopes
Aluminum	27	
Antimony	121	
Arsenic	75	77 [a], 83 [a]
Barium	137 [**]	135
Beryllium	9	
Bismuth (IS)	209	
Cadmium	111 [**]	106 [a], 108 [a], 114
Calcium	44	43, 88 [a]
Chlorine [a]		37 [a], (77, 82) [a]
Chromium	52	53, 54
Cobalt	59	
Copper	65	63 [**]
Erbium (dilution std) [e]	166	
Germanium (IS) *	72	
Indium (IS)	115	113, 118 [a]
Iron [f]	56	57, 54, 58
Lead [b]	208, 207, 206	204
Lithium (IS)	6	
Magnesium	24	25, 26
Manganese	55	
Molybdenum	98	97, 96, 92, 94, (108), 99[a]
Nickel	60	58, 62, 61, 64
Potassium	39	
Scandium (IS)	45	
Selenium [c f]	78	80, 78, 76, 77, 74
Silver	107	109
Sodium	23	
Terbium (IS)	159	
Thallium	205	
Thorium [d]	232	
Tin	118	
Uranium [d]	238	
Vanadium [c]	51	238, 50
Yttrium (IS)	89	
Zinc	66	64, 68, 67, 70

Method Modifications:
The internal standard Germanium (*) is not listed in either 6020 or 200.8 but is recommended by the instrument manufacturer to improve the recovery of the difficult-to-ionize elements (As, Cd, Se, Zn) in the presence of high concentrations of easily ionized elements such as Na and K.

A double-asterisk or underscore indicates the isotope suggested by the method by 200.8 (**) or 6020 (_) that is not used by C&T for quantitation.

[a] These masses are used for interference correction.

[b] Lead is quantitated using the sum of m/z 206, 207, and 208 to compensate for any differences in the abundances of these isotopes between samples and standards. The Agilent software includes the calculation based on all three isotopes, but reports them as mass 208.

[c] Target analyte listed in 6020A (Update 4, 1998 Draft) but not in 6020 (1994 version).

[d] Target analyte listed in 200.8 but not in either version of 6020.

[e] Erbium is the dilution standard, used by the data system to apply the correct instrument (auto-) dilution factor.

[f] MET-06 & MET-16: Hydrogen is used in the octopole to eliminate ArAr (m/z 78) and ArO (m/z 56) interference on masses 56 (Iron) and 78 (Selenium).

APPENDIX_4: INTERNAL STANDARD ASSIGNMENTS

The isotope used for target element quantitation must be within 50 mass units of the associated internal standard (6020 Section 5.4).

MET-06 & MET-16 (Agilent 7500ce)

Lithium (6)	**Scandium (45)**	**Germanium (72)**	**Yttrium (89)**
Beryllium	Aluminum	Arsenic	Molybdenum
	Calcium	Copper	
	Chromium	Selenium	
	Cobalt	Zinc	
	Iron		
	Magnesium		
	Manganese		
	Nickel		
	Potassium		
	Sodium		
	Vanadium		

Indium (115)	**Terbium (159)**	**Bismuth (209)**
Antimony	Barium	Thallium
Cadmium	Lead	
Erbium (dilution std)		
Silver		

Notes:
The lithium is enriched with ^{6}Li to reduce interferences from native lithium in the samples. The other internal standards are elements that are not typically encountered in environmental samples.

Method Modification: Curtis & Tompkins uses the internal standards recommended by the instrument manufacturer (Agilent). This mix meets the method criterion that the isotope used for sample quantitation is within 50 amu of the associated internal standard, but improves method performance by reducing matrix interferences.

C&T ISTD List:	^{6}Li, ^{45}Sc, ^{72}Ge, ^{115}In, ^{159}Tb, ^{209}Bi
6020 Recommends:	^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, ^{209}Bi
200.8 Recommends:	^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, ^{175}Lu, ^{209}Bi

APPENDIX_5: REAGENTS & STANDARDS PREPARATION

The standards and reagents listed below are those in use at the time this procedure was written. Alternate supplies may be used so long as they are of equivalent quality and all calibration, quality control, and traceability requirements are met.

REAGENTS

The preparation of all liquid or solid reagents, including dilutions into DI water, must be documented in the reagent prep benchbook. Each reagent is assigned a unique ID, based on the manufacturer and the date prepared. This ID is then recorded in the digestion benchbook each time the reagent is used.

Label each reagent with the reagent ID, concentration, prep date, and expiration date. All reagents should be prepared and stored in freshly cleaned glassware. Expired, discolored, or contaminated reagents should be discarded and the bottle cleaned before reuse.

Deionized water, ASTM Type II (ASTM D1193)
Argon, Purity: 99.99%
Air, C&T house compressed air
He, compressed, Purity: 99.9999%
H_2, compressed, Purity: 99.9999%

Aqua Regia: *prepare daily*
Prepare immediately before use in a glass bottle by adding 3 volumes of concentrated HCl to one volume HNO_3, typically preparing about 125mL aqua regia per batch (32mL HCl + 96mL HNO_3 => 128mL total). Aqua Regia must be prepared daily.

Nitric Acid (HNO_3), concentrated, InstraAnalyzed grade
JT Baker catalog # 7697-37-2
Store unopened bottles in the corrosives cabinet and open bottles under the fume hood for up to two years.

Hydrochloric Acid (HCl), concentrated, InstraAnalyzed grade
JT Baker catalog # 7647-01-0
Store unopened bottles in the corrosives cabinet and open bottles under the fume hood for up to two years.

STANDARDS

All source standards must be NIST-traceable and be documented in LIMS upon receipt, through the Standards Menu. The LIMS S-name is unique to the vendor that the source is obtained from; if a source standard is obtained from a different vendor, a new S-name must be assigned and the information entered in the “Standard Entry” table before the standard can be assigned a unique S# (standard number).

The LIMS Standard Maintenance database includes the catalog#, lot#, expiration date, and concentration of the standards as they are received from the vendor. Write the S# and the date received on both the standard vial and on the 'Certificate of Analysis' that accompanied the standard. If the supplier did not provide a certificate, call and request that a copy be faxed to C&T. The Certificate of Analysis must be kept on file in the appropriate binder.

Prepare working standards by diluting source standards to volume in a Class-A volumetric flask. Document the preparation in the standards benchbook. Enter the prep information into LIMS through the "Standard Maintenance" menu; LIMS will then assign a unique S# to that standard. Write the LIMS S# and expiration date in the benchbook along with the prep information. Label the standard vial with the contents, the LIMS S#, the expiration date of the standard, and the prep chemist's initials.

If the Certificate of Analysis or bottle label did not include an expiration date, assign an expiration date of one year from the date received.

Store standards at room temperature, away from light, to prevent photo-induced precipitation of silver. Working standards expire after 90 days except ICS-AB, which must be made weekly (Method 6020 requires a short expiration time because silver falls out of solution).

CALIBRATION BLANKS

Prepare calibration blanks (ICB/ CCB) daily by adding 1.0 mL concentrated HNO_3 and 1.0 mL HCl to DI water and diluting to 100 mL.

INTERNAL STANDARD SOLUTION

Source Standard (10ppm ^{6}Li, ^{45}Sc, ^{72}Ge, ^{89}Y, ^{115}In, ^{159}Tb, ^{209}Bi)
Agilent Catalog# 5183-4681

1ppm Working Standard: Make a 1:10 dilution of the Source Standard in 1%HCl and 1%HNO_3

TUNE SOLUTION

The Tune Solutions are made from the following source standards containing individual elements at 1,000ug/mL:

Ce	Cerium, SepxCertiprep Catalog # PLCE2-2X
Co	Cobalt, SepxCertiprep Catalog # PLCO1-2X
Li	Lithium, SpexCertiprep Catalog # PLLI1-2X
Tl	Thallium, SpexCertiprep Catalog # PLTL2-2X
Y	Yttrium, SpexCertiprep Catalog # PLY2-2X

MET-06 & MET-16 (Agilent 7500ce): 10 ug/L Ce, Co, Li, Tl, Y – Final Concentration

Make an acidified (1% HNO_3 plus 1% HCl) 10,000 ug/L intermediate:

1.) Add about 500mL deionized water to a 1L Class-1 volumetric flask.
2.) Carefully measure 10mL of nitric acid into the volumetric flask then swirl to mix.
3.) Measure 10mL of hydrochloric acid into the volumetric flask then swirl to mix.
4.) Using a calibrated autopipette, add 10mL of each (Ce, Co, Li, Tl, Y) 1,000 ug/mL standard to the flask.

5.) Swirl to mix then fill to the mark with DI water.
6.) Cap & invert 3 times to mix, allowing sufficient time for mixing with each inversion.

From this intermediate, make an acidified 10 ug/L solution:

7.) Add about 500mL deionized water to a second 1L Class-1 volumetric flask.
8.) Carefully measure 10mL of nitric acid into this volumetric flask then swirl to mix.
9.) Measure 10mL of hydrochloric acid into this volumetric flask then swirl to mix.
10.) Using a calibrated autopipette, add 1.0 mL of the 10,000 ug/L intermediate to the flask
11.) Swirl to mix then fill to the mark with DI water.
12.) Cap & invert 3 times to mix, allowing sufficient time for mixing with each inversion.
13.) Store at room temperature for up to 1 year.

TUNING CHECK STANDARDS

6020 Source Standard:	10,000 µg/L of Li, Co, In, Tl LIMS S-Name: 6020TS Inorganic Ventures Cat.# 6020TS
6020 Working Standard:	10 µg/L of Li, Co, In, Tl LIMS S-Name: ICPMS TUNE Dilute 0.05 mL of the 6020TS source standard into 50 mL of acidified (1% HNO3 plus 1% HCl) deionized water. This standard expires after 3 months.
200.8 Source Standard:	10,000 µg/L of Li, Co, In, Tl LIMS S-Name: 2008TS Inorganic Ventures Cat.# 2008TS
6020 Working Standard:	10 µg/L of Li, Co, In, Tl LIMS S-Name: 200.8 TUNE Dilute 0.05 mL of the 200.8 source standard into 50 mL of acidified (1% HNO3 plus 1% HCl) deionized water. This standard expires after 3 months.

CALIBRATION STANDARDS

Source Standards:

ICPMS ICS3A Spex Cat # ZCTB-6-500	1,000 ug/mL of Ca, Fe, Mg, K, Na, Al 10 ug/mL of Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, V, Zn 5 ug/mL of Tl
ICPMS ICS3B: (Spex Cat # ZCTB-7-500)	100 ug/mL of Sn, B, Ti 10 ug/mL of Sb, Mo

Working Standards: Dilute the following volumes of the source standard (ICS) or the indicated working standard into 50mL acidified (1% HCl plus 1% HNO_3) deionized water to make standards at the listed levels. These calibration standards expire after 30 days.

	Element	ICPMS CS1 (ug/L)	ICPMS CS2 * (ug/L)	ICPMS CS3 (ug/L)	ICPMS CS4 (ug/L)	ICPMS CS5 (ug/L)	ICPMS CS6 (ug/L)	ICPMS CS7 (ug/L)
Volume (mL) of Source		0.050 CS5	0.125 CS5	0.25 CS5	0.50 CS5	5.0 CS5	0.50 ICS3A&B	1.0 ICS3A&B
Sb	Antimony	0.10	0.25	0.50	1.0	10.	100	200
As	Arsenic	0.10	0.25	0.50	1.0	10.	100	200
Ba	Barium	0.10	0.25	0.50	1.0	10.	100	200
Be	Beryllium	0.10	0.25	0.50	1.0	10.	100	200
Cd	Cadmium	0.10	0.25	0.50	1.0	10.	100	200
Cr	Chromium	0.10	0.25	0.50	1.0	10.	100	200
Co	Cobalt	0.10	0.25	0.50	1.0	10.	100	200
Cu	Copper	0.10	0.25	0.50	1.0	10.	100	200
Pb	Lead	0.10	0.25	0.50	1.0	10.	100	200
Mo	Molybdenum	0.10	0.25	0.50	1.0	10.	100	200
Ni	Nickel	0.10	0.25	0.50	1.0	10.	100	200
Se	Selenium	0.10	0.25	0.50	1.0	10.	100	200
Ag	Silver	0.10	0.25	0.50	1.0	10.	100	200
Tl	Thallium	0.10	0.25	0.50	1.0	10.	100	200
V	Vanadium	0.10	0.25	0.50	1.0	10.	100	200
Zn	Zinc	0.10	0.25	0.50	1.0	10.	100	200
Al	Aluminum	1.0	2.5	5.0	10.	1,000	10,000	20,000
Ca	Calcium	1.0	2.5	5.0	10.	1,000	10,000	20,000
Fe	Iron	1.0	2.5	5.0	10.	1,000	10,000	20,000
Mg	Magnesium	1.0	2.5	5.0	10.	1,000	10,000	20,000
Mn	Manganese	0.1	0.25	0.5	1.0	100	100	200
K	Potassium	1.0	25.	50.	100	1,000	10,000	20,000
Na	Sodium	1.0	25.	50.	100	1,000	10,000	20,000

* ICPMS CS2 was the low point for an earlier model of ICP-MS and is not typically used for MET-06 and MET-16.

ICV / CCV

Source Standards:

ICVMS4A
Inorganic Ventures
Catalog# Z-MEB222149

1,000 ug/mL of Al, Ca, Fe, K, Mg, Na
10 ug/mL of Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, V, Zn
5 ug/mL of Tl

ICVMS2A
Inorganic Ventures
Catalog# Z-MEB222148

100 ug/mL of B, Sn, Ti
10 ug/mL of Mo, Sb

Working Standards: Combine 0.5mL of each source standard then bring to 50mL with acidified (1% HCl plus 1% HNO_3) deionized water to make the ICV/CCV standard. The calibration standards expire after 30 days.

	LIMS Name:	6020ICV1 (ug/L)	6020CCV1 (ug/L)
Ag	Silver	100	100
Al	Aluminum	100	100
As	Arsenic	100	100
Ba	Barium	100	100
Be	Beryllium	100	100
Ca	Calcium	10,000	10,000
Cd	Cadmium	100	100
Co	Cobalt	100	100
Cr	Chromium	100	100
Cu	Copper	100	100
Fe	Iron	10,000	10,000
K	Potassium	10,000	10,000
Mn	Manganese	100	100
Mg	Magnesium	10,000	10,000
Mo	Molybdenum	10,000	10,000
Na	Sodium	100	100
Ni	Nickel	100	100
Pb	Lead	100	100
Sb	Antimony	100	100
Se	Selenium	100	100
Tl	Thallium	50	50
V	Vanadium	100	100
Zn	Zinc	100	100

Note: Although the ICV and CCV are identical in composition, they are prepared and assigned separately because LIMS applies different rules to the instrument sequence.

ICS-A and ICS-AB

Stock Standards:
Inorganic Ventures, Catalog# 6020ICS-9A, contains interferent elements
Inorganic Ventures, Catalog# 6020ICS-9B, contains other elements

Working Standards: Prepare monthly by diluting the source standards or more frequently if low silver recoveries are observed.

ICS-AB (LIMS Name: ICPMSICSAB):
Using a 50mL volumetric flash, dilute 1.0mL HCl, 1.0 mL HNO_3 and 5.0 mL of the 6020ICS-9A source standard in deionized water.

ICS-AB (LIMS Name: ICPMSICSAB):
Using a 50mL volumetric flask, dilute 1.0mL HCl, 1.0 mL HNO_3 and 5.0 mL of the 6020ICS-9A source standard and 0.05 mL of the 6020ICS-9B source standard in deionized water.

Element		ICPMSICSA (mg/L)	ICPMSICSAB (mg/L)
Aluminum	Al	100	100
Calcium	Ca	300	300
Carbon	C	200	200
Chloride	Cl	2,121.5	2,121.5
Iron	Fe	250	250
Magnesium	Mg	100	100
Molybdenum	Mo	2.0	2.0
Phosphorous	P	100	100
Potassium	K	100	100
Sodium	Na	250	200
Sulfur	S	100	100
Titanium	Ti	2.0	2.0
Arsenic	As	0	0.10
Cadmium	Cd	0	0.10
Chromium	Cr	0	0.20
Cobalt	Co	0	0.20
Copper	Cu	0	0.20
Manganese	Mn	0	0.20
Nickel	Ni	0	0.20
Selenium	Se	0	0.10
Silver	Ag	0	0.05
Vanadium	V	0	0.20
Zinc	Zn	0	0.10

APPENDIX_6: TUNING & CALIBRATION CRITERIA

TUNING

The mass spectrometer must be "tuned" at the beginning of each sequence to verify the sensitivity, resolution, and mass alignment of the detector.

For MET-06 & MET-16, the Tuning Solution contains 100 ug/L Cerium (Ce), Cobalt (Co), Lithium (Li), Thallium (Tl) and Yttrium (Y). The following guidelines should be met before any analyses are performed:

6020 & 200.8 Mass Calibration	± 0.1 amu
6020: Resolution at 10% peak height	< 0.75 amu
200.8: Resolution at 5% peak height	< 0.75 amu
Oxide Ratio (Cerium Oxide: Cerium)	< 1%
Double-Charge Ions (m/z 140:70 ratio)	< 3%

		MET-06 & MET-16
Sensitivity	Li (m/z 7)	> 10,000 counts
	Co (m/z59)	> 20,000 counts
	Y (m/z 89)	> 20,000 counts
	Tl (m/z 205)	> 20,000 counts
Hydrogen-Mode Sensitivity	Co (m/z 59)	> 5,000 counts
	Se (m/z 78)	< 10 counts
	Fe (m/z 56)	Confirm Fe presence
	FeH:Fe (ratio m/z 57 to m/z 56)	~ 3%
Helium-Mode Sensitivity	Co (m/z 59)	> 5,000 counts
	ArCl or false As (m/z 75)	< 10 counts
	ClO or false V (m/z 51)	< 100 counts
	Ratio (m/z 51 to m/z 59)	< 1%

EPA 200.8 *Method Modification:* The tuning solution described in EPA 200.8 includes only Magnesium (m/z 24, 25 & 26) and Lead (m/z 206, 207 & 208). C&T uses the tuning solution described in 6020, because it includes lighter elements (Li^6) and additional mid-range elements.

6020 Tune Check Standard (ICPMS TUNE) contains 10 ug/L of Li (m/z 7), Co (m/z 59), In (m/z 115), and Tl (m/z 205). This standard must be analyzed at the beginning of the analytical sequence (and at the beginning of each 12-hour shift) to demonstrate that the detector hardware is correctly calibrated; no samples or calibration standards may be analyzed until the tune has passed acceptance criteria.

Frequency:	Beginning of 12-hour shift and every 12 hours
Precision:	≤ 5% RSD over 4 exposures
Resolution:	< 0.9 amu @ 10% Peak Height
Mass Calibration:	± 0.1 amu

200.8 Tune Check (200.8 TUNE) contains 10 ug/L of Be, Co, In, Mg, Pb. This standard must be analyzed at the beginning of the analytical sequence in which samples submitted for EPA 200.8 are run (and at the beginning of each 12-hour shift) to demonstrate that the detector hardware is correctly calibrated; no samples or calibration standards may be analyzed until the tune has passed acceptance criteria.

Frequency:	Beginning of 12-hour shift and every 12 hours
Precision:	≤ 5% RSD over 5 exposures
Resolution:	< 0.1 amu @ 5% Peak Height
Mass Calibration:	± 0.1 amu

6020 & 200.8 Method Modification: The EPA methods only require a tune check standard at the beginning of each sequence however C&T analyzes a tune check standard at the beginning of each 12-hour "tune shift" to standardize Mass Spectrometry criteria across the lab.

INTIAL CALIBRATION

An initial calibration curve, consisting of a minimum of a blank and five standards, is analyzed at the beginning of each sequence, after the instrument has passed the tune check criteria and before any samples are analyzed. The standards must be analyzed in order from lowest concentration (ICPMS CS1 or CS2) to highest concentration (ICPMS CS7); the lowest standard must be at or below the reporting limit for each element and the highest standard sets the top of the quantitation range. If one standard was obviously a bad injection, that standard may be reanalyzed immediately following the original curve.

Correlation coefficient: ≥ 0.995

Note: The correlation coefficient criterion is an in-house limit, since none of the methods specify any criteria.

CALIBRATION VERIFICATION (ICV/ CCV)

The initial calibration verification (ICV) standard is obtained from a second manufacturer (or a second lot# obtained from the same manufacturer as the calibration standard) and is used to demonstrate that the calibration standard was made up correctly and that the instrument

calibration. The recoveries for all requested elements must be within the acceptance limits before any samples are analyzed.

ICV Frequency:	Beginning of each sequence.
Recovery Limits:	90-110%.

The continuing calibration verification (CCV) standards are analyzed to demonstrate that the instrument calibration is still valid. The concentration of the CCV standards must be varied within the calibration range, excluding the highest and lowest levels (NELAC Ch.5.9.5.2.2.b requirement). The recoveries for the requested elements must be within the acceptance limits or the instrument must be recalibrated and any samples analyzed since the last passing CCV reanalyzed.

CCV Frequency:	After every 10 samples and end of sequence.
Recovery Limits:	90-110%.

CALIBRATION BLANKS (ICB/CCB)

An initial calibration blank (ICB) verification must be run at the beginning of each analytical run, with the ICV. A continuing calibration blank (CCB) verification must be analyzed (with each bracketing CCV), after every 10 analytical samples, including batch QC samples, and at the end of the sequence. Target elements should not be detected in the CCB at any level above 3 times the Instrument Detection Limit (IDL).

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the ICB/CCB must be ND > 2x MDL.

INTERFERENCE CHECK STANDARDS (ICS)

Interference Check Standards contain known concentrations of interfering elements and are used to demonstrate the magnitude of the interferences and that the inter-element correction factors correctly compensate for these interferences. These standards are analyzed at the beginning, every 12 hours, and end of every sequence. The recoveries for the requested elements must be within the acceptance limits or the instrument must be recalibrated and any samples analyzed since the last passing CCV reanalyzed.

Chloride is included in the ICS to evaluate software corrections for chloride-related interferences. Iron is used to verify that the resolution of the spectrometer is adequate for quantitation of manganese. Molybdenum evaluates oxide effects on cadmium isotopes.

ICS-A contains only the elements that typically cause interferences when present at relatively high concentrations. The affected elements (which are not included in this standard) should not be observed at levels above the reporting limit.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the measured concentrations of any elements that are not included in the ICS-A standard must be ND > 2x MDL.

The AFCEE (Air Force) Model QAPP, Version 3.0, specifies that the elements that are not added to the ICS-A solution must not be detected at levels above the reporting limits, however according to Agilent and SPEX, low levels of some elements (notably chromium) will be present in the ICS-A due to impurities that cannot be removed from the vendors' cation sources. C&T controls only those elements to which Inter-element correction factors are applied.

ICS-AB contains the ICS-A elements at relatively high levels and all other target elements at lower levels (typical of those seen in environmental samples). None of the EPA ICP-MS methods (200.8, 6020, or 6020A) specify control limits for this standard, so C&T has chosen to standardize the ICP methods by using the EPA 6010 limits.

Recovery Limits: 80-120%

Method Modification: C&T requires that the ICSAB be re-analyzed (and pass acceptance criteria) at the end of each analytical sequence and pass the acceptance criteria listed above; if the standard fails, any samples analyzed for the failing element after the last passing ICS-AB must be reanalyzed. The limits listed above are those typically required in DoD project plans and have been adopted by C&T because method 200.8 does not require these interference check standards and EPA methods 6020 and 6020A do not specify recovery limits for this standard and the instrument manufacturer (Agilent) recommends it "for informational purposes only".

APPENDIX_7: USING CHEMSTATION FOR INSTRUMENT CONTROL

Instrument Control Diagram
Diagram displays a schematic of the instrument, allowing you to check the gauges, meters, and ON/OFF switches. Instrument control is accessed through this diagram.

Instrument >> Instrument Control

Meter Control Diagram
Diagram displays additional details for specific components (vacuum, water, gases, plasma Rf, housing, S/C) associated with instrument control.

Instrument >> Instrument Control
Meters >> Meter Control

Tune Panel
Tune Panel displays detailed information regarding system parameters.

Instrument >> Tune…

'Standby' Mode to 'Analysis' Mode
The instrument will go to 'Standby' mode, after maintenance, when the vacuum has pumped down and is ready. Then turn on the instrument by:

Select *Instrument >> Instrument Control*.
Plasma >> Plasma On
YES

'Standby' Mode to 'Shutdown' Mode
To do any maintenance in the vacuum chamber, the plasma must be turned off and the vacuum shut down.

Select *Instrument >> Instrument Control.*
Plasma >> Plasma Off
YES
Vacuum >> Vacuum Off

'Shutdown' Mode to 'Standby' Mode
Open ICPMS TOP program,
Select: *Instrument >> Instrument Control*
Vacuum >> Vacuum On
YES

Allow between 15 minutes to 2 hours to pump the vacuum down to $5x10^{-4}$ kPa.
Once the instrument has reached the 'Standby' Mode:

Verify that the instrument exhaust rate is correct (5 m3/min).
Verify that the water chiller is turned on.
Verify that the Argon pressure is correct (700 + 20 kPa).

Turn Off the Instrument
To do any major maintenance (on the cones, extraction lenses, source, etc.), the plasma must be turned off and the vacuum shut down.

Put the instrument into 'Standby' Mode as described above.
Turn off the power switch on the front panel.
Turn off the rotary pump power switches on instrument.
Turn off the main power breaker on the back of the instrument.
Turn off the rotary pump power switch on the actual pump.

MET-06 & MET-16 (Agilent 7500ce) GENERAL STARTUP & ANALYTICAL PROCEDURE ___

Restart the MET-06, or MET-16, Instrument from Overnight Stand-by Mode

1.) Replace the tubing around the peristaltic pumps.
2.) Refill the DI rinse-water bottle with acidified (1% HCl plus 1% HNO_3) DI water.
3.) Turn on the computer then click the ICPMS TOP icon.
4.) Open the Instrument Control Diagram.
5.) Verify that the instrument is in 'Standby' mode.
6.) Click YES when asked to verify the tubing connection; this will turn on the plasma and warm up the instrument. The warm-up will take approximately 5 minutes and the instrument icons will turn green as each system reaches 'ready'.

Tune the MET-06, or MET-16, Instrument:

7.) Open Tune Panel.
8.) Select: nogas.u from the dropdown menu.
9.) Select: ALS >> Go To >> '2'(Tune Solution).
10.) Select: Tune >> Sensitivity, then START.
11.) Verify that the RSD and Counts results are in the correct range.
Note: RSD's should be <5%, not to exceed 10%.
12.) Verify that the height of the Li^{6+} peak is less than ½ the Li^{7+} peak; once it reaches approximately ½, preventative maintenance (cleaning the cones) should be scheduled.
13.) Click STOP.

14.) Select: File >> Save Tune Values…(save to nogas.u)
15.) Select: H2.u from the dropdown menu
16.) Select: Tune >> Sensitivity, then START..
17.) Verify that the RSD and Counts results are in the correct range
18.) Click STOP

19.) Select: File >> Save Tune Values…(save to H2.u)

20.) Select: He.u from the dropdown menu
21.) Select: Tune >> Sensitivity, then START
22.) Verify that the RSD and Counts results are in the correct range
23.) Click STOP

24.) Select: File >> Save Tune Values…(save to He.u)
25.) Select: nogas.u from the dropdown menu
26.) Select: Tune >> Oxide Ion, then START.
27.) Verify that the oxide ratio is ≤ 1.0%.
28.) Click STOP.

29.) Select: Tune >> Doubly Charged Ion, then START.
30.) Verify that the ratio is ≤ 3%.
31.) Click STOP.

32.) Select: Tune >> Resolution/ Axis button, then START.
33.) Verify that the 'Axis' reading is within ± 0.1 amu from the true value for each element.
34.) Verify that the peak width (W-10%) is between 0.7 - 0.8 (must be < 0.9).
35.) Click STOP.

36.) Select: File >> Generate Multi-mode Report.
37.) Verify that all of the listed parameters are within acceptance limits.

Analyze the Pulse/ Analog Standard on MET-06 or MET-16:

38.) Select: ALS >> Go To >> rinse
39.) Select: ALS >> Go To >> '5'(50ppb P/A Factor solution)
40.) Select: Tune >> P/A Factor
41.) Click 'Load Masses from aqu. Method'
42.) Click RUN
43.) After P/A Factor is RUN, Chemstation asks, '"Would you like to adopt new P/A Factors? (Refer to the report displayed on notepad.)"
44.) Observe intensities. Click 'Yes'
Note:If nessessary, adjust tuning parameters and reanalyze P/A Factor solution to aquire ,at a minimum, all of the internal standards
45.) Select: File >> Print, then close the screen.
46.) Select: File >> Copy Tune Parameters…
47.) Select: Export to button.
48.) Click Browse…, Select H2.u
49.) Check the Detector Parameters and P/A Factors box
50.) Click Copy
51.) Click Browse…, Select He.u
52.) Check the Detector Parameters and P/A Factors box
53.) Click Copy
54.) Click Close

APPENDIX_8: INTERFERENCES

Polyatomic Interferences occur when an isotope of an interferent occurs at the same mass as the primary mass of an element of interest. Corrections can be calculated, based on the *observed* ratio of isotopes in a standard. See Section 3.2 of SW-846 method 6020 for further details and example equations.

Specific interferences which have been identified in the referenced methods include $ArCl^+$ with the ^{75}As signal and MoO^+ with the Cd isotopes.

MET-06 & MET-16: For the Agilent 7500ce, most isobaric molecular interferences are reduced by the octopole reaction system running in Hydrogen Mode or Helium Mode.

Memory Interferences, also called 'carry-over', may occur when a low-level sample is analyzed immediately after a high-level sample. These interferences are minimized by rinsing the system between all samples and standards.

Physical Interferences are typically those occurring during the digestion, nebulization, and transport. Common physical interferences include:

1. - Loss of volatile elements (antimony) during the digestion process if the digestate is superheated or allowed to go dry during heating.
2. - Precipitation of certain elements (silver) during the digestion process if present in relatively high levels.
3. - Zinc contamination due to dusty surroundings.
4. - Chromium contamination in purchased ICS-A standards (a problem for all ICS suppliers – per Agilent Technologies).
5. - Differences in sample viscosity and surface tension due to high levels of dissolved solids. Serial dilutions may be used to identify this type of interference, which may clog the nebulizer and tubing.

This method includes the use of internal standards, which minimize the effects of these interferences on sample results by closely matching the physical properties of the associated internal standard to those of the target elements.

APPENDIX_9: INSTRUMENT CONDITIONS

MET-06 & MET-16 (Agilent 7500ce)

Argon	Purity (%):	99.99	
	Pressure (kPa):	700 ± 20	(Concentric Nebulizer)
	Flow Rate (L/min):	1.2	
Helium:	Purity (%):	99.9999	
Hydrogen:	Purity (%):	99.9999	
Water Cooler	Temperature (°C):	Set Point: 20°C (acceptable between 15°C to 25°C)	
	Pressure:	40 psi	
	Flow Rate:	WC/IF: 1.1 to 2.0 RF/TP: 1.1 to 3.0	
MSD Vacuum	Pressure (kPa):	5×10^{-4}	
Exhaust Flow	Rate (m^3/min):	5	

Quantitation Channels (Octopole Reaction System):

Hydrogen Mode (LIMS Channel H):	Fe, Se
Helium Mode (LIMS Channel E):	As, Cr, Co, Cu, Mn, Ni, Na, V, Zn
Vacuum only (LIMS Channel A):	Al, Sb, Ba, Be, Cd, Ca, Pb, Mg, Mo, K, Ag, Tl

APPENDIX_10: MAINTENANCE & TROUBLE-SHOOTING

ROUTINE MAINTENANCE

Any maintenance performed on the instrument must be documented in the instrument maintenance logbook. Whenever preventative or trouble-shooting maintenance is performed, document:

1.) the reason the maintenance was necessary,
2.) the action taken, and
3.) the resolution of the maintenance ("passed CRI", "RSD's OK", etc.).

Each benchbook entry must be initialed and dated by the analyst performing the maintenance. If an outside service contractor performs the maintenance, the identity of the contractor's company and a summary of the maintenance performed should be documented in the benchbook.

Detailed instrument maintenance instructions are described in Chapter 4 of the Agilent 7500 Hardware Manual. Maintenance and part numbers for the Integrated Sample Introduction System (ISIS) are described in the ISIS Manual.

Peristaltic Pump Tubing should be replaced whenever the tubing becomes yellow, discolored, or cracked; typically the sample tube and ISTD tubing should be changed daily and the rinse vessel tube (drain tube) every month. High RSD's in the tuning mode typically demonstrate that peristaltic sample and ISTD pump windings need to be replaced. The type of tubing can be identified by the color-coded tabs fixed to the piece. Turn off the peristaltic pump, then replace the tubing. Order replacement tubing from CPI and keep at least one spare set on-hand.

Sample Tube	Tygon (Orange-Orange, 0.89mm ID)	CPI Cat # 4062-535
ISTD Tube	Tygon (Orange-Red, 0.19mm ID)	CPI Cat # 4062-5008
Drain Tube	PharMed (Yellow-Blue, 1.52mm ID)	CPI Cat # 4062-01916
ISIS Pump Tube	Tygon(White-White, 0.60ml/min)	CPI Cat # 4062-440

Soak Tygon tubing in acidified (5% HCl plus 5% HNO_3) deionized water for at least 2 hours. Verify that any contamination has been rinsed from the tubing by running an instrument blank.

The **nebulizer, spray chamber,** and **torch** may also need to be cleaned or replaced when elements begin routinely failing the RSD or CCV requirements. Allow the torch and interface to cool for at least 10 minutes before handling. Always wear gloves when handling the nebulizer and associated parts; if the surface becomes contaminated with oils, the signal will become unstable.

The **cones** may need to be cleaned or replaced when elements begin routinely failing the RSD or CCV requirements, or any of the following are observed: low response, distorted peak shape, or interface vacuum pressure >5 x 10^2 kPa. The sampling cone typically requires more frequent

maintenance than the skimmer cone, due to its proximity to the torch. See pages 4-59 to 4-66 of the Hardware Manual for complete details.

Caution: Do not touch the skimmer cone orifice or allow it to come into contact with other instrument parts, as even slight pressure can damage the orifice. If the orifice of either cone becomes damaged or distorted, the cone must be replaced.

Nickel Skimmer Cone	Agilent Cat# G1820-65050
Nickel Sampling Cone	Agilent Cat# G1820-65238

The **extraction lens** and **Einzel lens assembly** may need to be cleaned when the instrument cannot be tuned or the ion signal is unstable. *Always* wear clean powder-free latex or cotton gloves when handling any vacuum chamber parts.

Polishing Paper	Agilent Cat# G1833-65404

For MET-06 & MET-16 (Agilent 7500ce):
Only the Einzel lens assembly is inside the vacuum chamber. Reduce the maintenance time by first cleaning the Einzel lens assembly with specified Agilent polishing paper, reinstalling it (see pages 4-146 to 4-147 of the Hardware Manual) and beginning the vacuum pump-down, then cleaning the extraction-omega lens assembly in the same fashion. For the Agilent 7500ce, see pages 4-87 to 4-94 of the Hardware Manual for detailed instructions

Clean the o-ring with methanol and check for damage whenever re-installing the lenses; any dust or nicks in the o-ring will cause a vacuum leak when the instrument is pumped down.

The **Octopole Reaction System** in MET-06 (Agilent 7500ce) may need to be cleaned when the counts in the normal mode (nogas.u) do not recover instantaneously when switched from the hydrogen mode (h_2.u) to the normal mode.

1.) Remove the octopole from the instrument (see Hardware Manual pp. 4-97 to 4-99).
2.) Sonicate entire octopole assembly (NOT INCLUDING the cell upper case, cell lower case, or cell assembly screws) in deionized water for 5 minutes.
3.) Change deionized water and repeat sonication two more times.
4.) Dry the octopole with nitrogen gas.
5.) Remove the Plate Bias Lens (see Hardware Manual pp. 4-102 to 4-103).
6.) Clean the plate bias lens with specified Agilent polishing paper and deionized water.
7.) Dry with nitrogen gas.
8.) Reinstall octopole reaction system.

The **electron multiplier (EM)** normally operates in pulse mode but automatically switches to analog mode when analyzing high concentration samples. The EM should be changed when adjusting increasing the EM voltage to meet sensitivity requirements results in an Analog voltage of ~3300 or a Pulse voltage of ~1800. See pages 4-85 to 4-88 of the Hardware Manual for detailed instructions.

MET-06 Electron Multiplier	Agilent Cat# G1833-65420 or -65575

The **vacuum pump oil** should be changed every 6 months or if the pressure gauge reads 10 microns Hg or above. It typically takes 1-2 days after the oil change to pump the vacuum back down and remove all traces of oil from the system. See pages 4-95 to 4-97 of the Hardware Manual for detailed instructions.

Rotary Pump Oil	Agilent Cat# 6040-0834

The water **filter** for the re-circulator should be changed about every 6 months, or whenever the warning lights go on.

Water filter	Agilent Cat# G1820-65018

Lubricate the autosampler tracks approximately every six months by wiping the tracks with a Kim-Wipe saturated with 1-in-3 or clear oil.

TROUBLE-SHOOTING

For trouble-shooting purposes, the ICPMS can be separated into three distinct sections:
1.) The sample introduction system, from the autosampler sipper through the tubing and connections to the nebulizer and spray chamber and ending with the torch.
2.) The second consists of the cones back through the lenses.
3.) The third begins at the quadrupole (or octopole) and ends with the detector.

To begin trouble shooting, run a sequence of 5 sets of a high standard followed by a blank, while monitoring counts for two IS and two analytes. The counts for IS and high standard should not vary more than 5% between runs, and the IS should not drift more than 30% between the blank and high standard. If the IS counts are stable but the analyte counts drift or if the IS counts drift and the analyte counts are stable this indicates that there is problem in section one. Begin replacing and/or adjusting components in section one until this behavior discontinues.

If both counts for the IS and analyte are drifting through the run this indicates a problem in section two. Clean or replace components of section two until this behavior ceases.

To trouble shoot section three monitor drift of two plasma gases, such as mass 80 and 76 while aspirating no sample. A drift in counts of these readings indicate a problem with the Electron Multiplier or Mass Analyzer.

Problem: High RSD's in the tuning mode.
Solution: Peristaltic sample and ISTD pump windings need to be replaced.

Problem: Instrument cannot be tuned or the ion signal is unstable.
Solution: The extraction lens and Einzel lens assembly may need to be cleaned

Problem: Internal standard recoveries are stable for 1-2 hours after starting up instrument but then begin slowly rising and do not drop back to the baseline after repeated rinsing.

Solution: The plastic lens spacers in the source may have developed a charge. To check this possibility, go to the Tune screen voltages. Read the Tune Solution then change the voltages to zero. Let the instrument run for a few minutes to clear any temporary charge, then go back to the normal settings. Check the Tune Solution voltages - if the voltages change significantly, the spacers should be replaced.

This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

APPENDIX_11: CALCULATIONS

The Agilent software automatically performs internal standard correction on the analyte intensities. These corrected intensities are then used to determine the calibration curve and analyte concentrations.

Instructions for printing Uncorrected Intensities:

1.) Open Offline Data Analysis program.
2.) From the 'Tools' drop down menu, select 'Configure Do List…', select 'FullQ-Summary-Printer', Add to the Do List.
3.) From the 'Tools' drop down menu, select 'Do List'.
4.) Select 'FullQ-Summary-Printer'.
5.) Select file to be printed, 'Add file', then process file.
6.) Selected file will print to the associated printer.

The corrected intensities are calculated by dividing the area count of the target ion by the ratio of the counts in the internal standard divided by the concentration of the internal standard in the first level of the calibration curve. These equations, as described in the Agilent Operator Manual, are:

$$y = y_\sigma / (y_i / x_i) \qquad (y = y_\sigma \text{ if } x_i = 0)$$

Where: x_i = concentration of the internal standard in the first level of the calibration curve
y_i = count of the internal standard
y_σ = count of the target ion

Moisture Corrected Results
Dry Weight Concentration (ug/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

Using Linear Regression:
The corrected intensities are then used to calculate the following parameters from the initial calibration: slope (s) and intercept (I). The slope and intercept define a relationship between the concentration and instrument response of the form:

$$y = s\,x + I$$

Where: y = predicted instrument response
s = response slope
x = concentration of standard
I = intercept

Rearrangement of the above equation yields the concentration corresponding to an "on-column" instrumental measurement:

$$x = (y - I) / s$$

Where: x = calculated on-column concentration for a sample
y = actual instrument response for a sample
s and I are calculated slope and intercept from calibration.

Final sample concentration is then corrected for any sample prep dilution factors:

$$\text{Sample Conc (mg/L)} = x * pdf * idf$$

Where: x = calculated on-column concentration for a sample
pdf = prep dilution factor
idf = instrument dilution factor

BATCH QC CALCULATIONS

Percent Recovery (%R):
The recovery is the measured concentration divided by the true concentration of the spike.

$$\%Recovery = (Cf - Cs) / (Cws * Vws / S) * 100$$

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard
S = Sample weight or volume

Relative Percent Difference (RPD):
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

$$\%RPD = |(Cs - Cdup)| / ((Cs + Cdup)/2) * 100$$

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

For soil MS/MSD's where the sample weights are not weight-targeted, the expected concentrations will vary with sample weight (because the same volume of spike standard is being added to different weights of sample) and must be accounted for when calculating RPD:

$$\%RPD = |((Wms/Wmsd)*Cms - Cmsd)| / (((Wms/Wmsd) * Cms + Cmsd)/2) * 100$$

INITIAL CALIBRATION

Correlation Coefficient:
For each compound, the correlation coefficient for the initial calibration curve is calculated by:

$$\text{Correlation coefficient} = \frac{\sum_{i=1}^{n} (Yobs - Ymean)^2 - ((n-1)/(n-p)) * \sum_{i=1}^{n} (Yobs - Yi)^2}{\sum_{i=1}^{n} (Yobs - Ymean)^2}$$

Where:
Yobs = observed intensity for each ICAL std conc.
Ymean = mean observed intensity from the ICAL standards
Yi = calculated (or predicted) intensity for each ICAL std conc.
n = total number of ICAL points
p = number of adjustable parameters in equation (linear= 1, quadratic= 2)

First Order Coefficient (Slope) - For Linear Regression ICAL

$$\text{Slope} = \frac{n * \sum (Cx * Ax) - (\sum Cx) * (\sum Ax)}{n * \sum (Cx^2) - (\sum Cx)^2}$$

Where:
n = total number of ICAL points
Ax = Area or Intensity of the compound being measured
Cx = Concentration of the compound being measured

CALIBRATION VERIFICATION

$$\%\ \text{Difference}\ (\%D) = \frac{AVG\ RF - RFc}{AVG\ RF} * 100$$

Where:
AVG RF = Average response factor from initial calibration
RFc = Response factor from current verification check standard

$$\%\text{Drift}\ (\%D) = \frac{(C1-Cc)}{C1} * 100$$

Where:
C1 = Calibration Check Compound standard concentration
Cc = Measured concentration of CCC

STANDARDS PREPARATION

To determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where:
- Vss = Vol (mL) of Source Standard needed to make Working Standard
- Vws = Final Volume (mL) of Working Standard
- Cws = Final Concentration (ug/mL) of the Working Standard
- Css = Concentration (ug/mL) of the Source Standard

APPENDIX_12: EPA 200.8

The following criteria and discussion apply to any samples submitted for the EPA 200.8 analysis:

TUNING:
Method Modification: The Tuning Solution described in EPA 200.8 includes only Magnesium (m/z 24, 25 & 26) and Lead (m/z 206, 207 & 208). C&T uses the tuning solution described in 6020, because it includes lighter elements (Li^6) and additional mid-range elements.

C&T's Tuning Solution contains 100 ug/L Cerium (Ce), Cobalt (Co), Lithium (Li), Thallium (Tl) and Yttrium (Y). The following guidelines should be met before any analyses are performed:

6020 & 200.8 Mass Calibration	± 0.1 amu
6020: Resolution at 10% peak height	< 0.75 amu
200.8: Resolution at 5% peak height	< 0.75 amu
Oxide Ratio (Cerium Oxide: Cerium)	< 1%
Double-Charge Ions (m/z 140:70 ratio)	< 3%

		MET-06 & MET-16
Sensitivity	Li (m/z 7)	> 10,000 counts
	Co (m/z59)	> 20,000 counts
	Y (m/z 89)	> 20,000 counts
	Tl (m/z 205)	> 20,000 counts
Hydrogen-Mode Sensitivity	Co (m/z 59)	> 5,000 counts
	Se (m/z 78)	< 10 counts
	Fe (m/z 56)	Confirm Fe presence
	FeH:Fe (ratio m/z 57 to m/z 56)	~ 3%
Helium-Mode Sensitivity	Co (m/z 59)	> 5,000 counts
	ArCl or false As (m/z 75)	< 10 counts
	ClO or false V (m/z 51)	< 100 counts
	Ratio (m/z 51 to m/z 59)	< 1%

200.8 Tune Check (200.8 TUNE) standard is different than that used for 6020, containing 10µg/L of Be, Co, In, Mg and Pb. This standard must be analyzed at the beginning of the analytical sequence in which samples submitted for EPA 200.8 are run (and at the beginning of each 12-hour shift) to demonstrate that the detector hardware is correctly calibrated; no samples or calibration standards may be analyzed until the tune has passed acceptance criteria.

Frequency:	Beginning of 12-hour shift and every 12 hours
Precision:	≤ 5% RSD over 5 exposures
Resolution:	< 1 amu @ 5% Peak Height
Mass Calibration:	± 0.1 amu

6020 & 200.8 Method Modification: The EPA methods only require a tune check standard at the beginning of each sequence however C&T analyzes a tune check standard at the beginning of each 12-hour "tune shift" to standardize Mass Spectrometry criteria across the lab.

INTERNAL STANDARDS
EPA 200.8 requires a minimum of 3 internal standards and recommends the use of five; C&T uses the five discussed in the method (^{6}Li, ^{45}Sc, ^{89}Y, ^{115}In, ^{159}Tb, ^{209}Bi) plus ^{72}Ge, as recommended by the instrument manufacturer.

For 200.8, the Internal Standard recovery limits are 60-125%, or reanalysis and/or dilution for matrix interferences is required.

INITIAL CALIBRATION:
Method 200.8 only requires a 3-point calibration and does not specify a correlation coefficient (r) criteria but C&T uses the standard criterion of ≥0.995. The same general calibration criteria must be followed (ie: intermediate levels may not be dropped, the low point must be at or below the reporting limit, etc.). Additional details regarding the procedure and requirements are described in the main body of this SOP and in Appendix 6 above.

CALIBRATION VERIFICATION (ICV/CCV):
Method 200.8 calibration verification requirements match those in EPA 6020; see Appendix 6 for details.

INTERFERENCE CHECK STANDARDS (ICS-A and ICS-AB):
EPA 200.8 does not mention either of these standards; C&T standard practice is to include these standards in the analytical sequence, following SW-846 guidance as described in Appendix 6 above.

SAMPLE ANALYSIS:
EPA 200.8 requires that the samples be aspirated for at least 30 seconds prior to collection of data to allow the response to stabilize; C&T aspirates the samples for approximately 30 seconds or longer when necessary, as described in the main body of this document.

EPA 200.8 requires a minimum of 3 integrations for each mass, reporting the average of the three; this matches C&T standard practice as described in the main body of this SOP. The method does not discuss a limit on the RSD between these integrations; C&T has established an internal requirement of 20%RSD between the integrations. If the RSD is >20, the sample must be rerun and/or diluted to reduce matrix interferences.

EPA 200.8 requires that the instrument be flushed for 1 minute between samples; MET-06 and MET-16 automatically flush the system for approximately 90 seconds between samples and will go on to the next autosampler tube once the signal has stabilized.

BATCH QC:

- Method Blank: EPA 200.8 requires redigestion and reanalysis of any sample associated with a method blank containing laboratory contamination greater than **2.2x the MDL** for that element and <10x the concentration in the sample. If the sample is ND for the blank contaminant or is >10x the level found in the blank, the sample may be reported without corrective action.

- BS/BSD: EPA 200.8 specifies LCS (or LFB, "Lab Fortified Blank") recovery limits of **85-115%** but does not discuss the use of a duplicate. C&T digests and analyzes a pair of blank spikes with every batch to better monitor both the accuracy and precision of the process; RPD limits are based on statistically generated control limits.

- MS/MSD: EPA 200.8 specifies recovery limits of **70-130%** but does not discuss the use of a duplicate. C&T digests and analyzes a pair of matrix spikes with every batch to better monitor both the accuracy and precision of the process on real-world samples; RPD limits are based on statistically generated control limits.

- Serial Dilution recovery limits are 90-110%, matching the SW-846 requirement.

Curtis & Tompkins, Ltd.

Digestion & Analysis of
Aqueous Samples for
MERCURY

EPA 7470 / EPA 245.1

Approved by:

Porter Williams 3/9/12
Department Manager/ Date

Marilyn Brazelton 3/9/12
QA Director/ Date

Read & Understood by:

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Curtis & Tompkins, Ltd.

Digestion & Analysis of
Aqueous Samples for
MERCURY
EPA 7470 / EPA 245.1
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Digestion & Analysis of Aqueous Samples for
MERCURY
EPA 7470 / EPA 245.1

SCOPE

This SOP describes the sample digestion and analysis of ground water, surface water, drinking water, and leachate samples for the determination of mercury by CVAA. The reporting limit for this procedure is 0.2 µg/L.

In this procedure, organometallic compounds are broken down and converted to mercuric ions by digesting the sample with permanganate and persulfate under heated conditions. The concentration of mercury in the resulting digestate is then determined by atomic absorption. This procedure is not applicable to soil or solid waste samples.

REFERENCES

EPA 7470A, MERCURY IN LIQUID WASTE (MANUAL COLD-VAPOR TECHNIQUE), SW-846 Update 1, Sept.1994
EPA 245.1, Methods for Chemical Analysis of Water & Wastewater, EPA 600/4-79-020
Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Rev 4.2 October 2010

Additional SOP's and Guidance Documents:
NELAC Chapter 5, Quality Systems, June 2003
DoD Quality Systems Manual, Version 4.1, 4/22/09
DoE Quality Systems Manual, Version 2.2, Oct.2006
US ACOE "Shell" Document, EM 200-1-3, February 2001
C&T SOP QA 1.4, *Balance Calibration Check & Maintenance*
C&T SOP QA 1.5, *Calibrating & Maintaining Temperature Controls*
C&T SOP QA 1.6, *Pipet Calibration Check Procedures*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL)*
C&T SOP QA 8.4.1, *State Program Requirements*
C&T SOP QA 8.5.1, *DoD Program Requirements*
C&T SOP QA 8.6.1, *DoE Program Requirements*
C&T CS SOP 2.3, *Subsampling & Compositing*

SAMPLE PRESERVATION & HOLDING TIME

Preservative: HNO_3 to pH < 2. Store at 4°C (± 2°C)
Holding time: 28 days from sample collection until digestion & analysis

SAFETY

Mercury is a highly toxic element - take care not to inhale the vapor. This procedure uses concentrated nitric and sulfuric acids that *will* cause injury if allowed to contact skin or eyes.

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Gloves and safety glasses should be worn at all times when handling samples, standards, or reagents.

INTERFERENCES

Mercury is a relatively volatile element that may be vaporized if the digestion temperature is not closely monitored. Sulfide interferences are reduced through use of potassium permanganate in the digestion step.

QC REQUIREMENTS

Initial Calibration curves must be composed of a minimum of a blank and five standards, which are digested in the same manner as the associated samples. Instrument should be calibrated before each analytical sequence. The correlation coefficient must be $\geq$ 0.995.

Initial Calibration Verification (ICV) standards must be prepared from stock standards received from a source other than those used for the initial calibration curve. The ICV is analyzed prior to sample analysis with a recovery between 90-110%.

Initial Calibration Blank (ICB) is a reagent blank that follows the ICV. Mercury must not be detected above the MDL.

Continuing Calibration Verification (CCV) standards must be analyzed after every 10 samples, including batch QC samples, and at the end of the analytical sequence. The concentration of the CCV standard is at the mid-range of the calibration curve. The recovery for the CCV must be within 80-120%. If a CCV fails to meet the recovery criteria, the analysis is terminated, the instrument re-calibrated, and all samples analyzed after the last passing CCV reanalyzed.

Calibration Blanks (CCB) are reagent blanks that follow every CCV. Mercury must not be detected above the MDL.

Batch QC: The following quality control (QC) samples must be prepared in the same manner as the analytical samples at a rate of once per twenty or less samples. C&T in-house limits are based on control charts and are updated semi-annually; see the associated SOP *Mercury QC Limits, Table-1* for the current limits.

Method Blank (MB): Digest and analyze a method (prep) blank with each batch of 20 samples or less to demonstrate that the glassware and reagents are free of contamination.

Blank Spike / Blank Spike Duplicate (BS/BSD): Digest and analyze a blank spike and a blank spike duplicate for every batch of 20 samples or less to demonstrate that the procedure is accurate (measured by the *recovery*) and precise (measured by the *RPD*) in the *absence* of matrix interferences.

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Matrix Spike (MS) / Matrix Spike Duplicate (MSD): Digest and analyze an MS/MSD with every batch of samples to demonstrate the accuracy and precision of the process on real-world samples.

Note: Client- or project-specific quality assurance project plans (QAPPs) may require an MS/MSD in place of the SDUP and SSPIKE. For batches containing samples from these projects, the QAPP requirement takes precedence over this SOP.

Sample Interference Verifications: The method recommends that whenever a new or unusual matrix is encountered, a series of tests be performed to ensure that neither positive nor negative interferences are distorting the accuracy of the reported values. These are optional steps that should be implemented for any batch containing "Level 3" or Level 4" samples, as nearly all Department of Defense project plans include this requirement. These should also be analyzed whenever the analyst suspects that sample viscosity, salt content, or other matrix interferences are likely.

Serial Dilutions: Analysis of a 5x dilution should agree within ± 10% (90-110% recovery) of the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference should be suspected.

Post Digest Spikes: An analyte spike added to a portion of a prepared sample digest, or its dilution, should be recovered to within 75-125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits a matrix effect should be suspected.

Method Detection Limit (MDL): MDL studies are performed when major changes to instruments or methods occur, in order to demonstrate that the sample preparation and analysis procedures are adequate to meet required reporting limits. The MDL is determined by digesting and analyzing at least 7 replicates of a low-level blank spike, determining the standard deviation and applying a multiplier. See the QA SOP "Determining MDLs" for details.

Instrument Detection Limit (IDL): The IDL is determined by analyzing seven instrument blanks on three non-consecutive days. The standard deviation of the calculated concentrations is determined for each day; the IDL is the sum of the three standard deviations.

Limit of Detection (LOD) must be determined quarterly for DoD and annually for NLEAC compliance and is based on a laboratory control sample (MDL check sample) that is spiked 2 to 4 times the MDL, digested and analyzed on every instrument. See the QA SOP for Method Detection Limits for details.

Limit of Quantitation (LOQ) must be determined quarterly for DoD and annually for NELAC and is based on a laboratory control sample that is spiked 1 to 2 times the reporting limit. It is only analyzed once per method. See the QA SOP for Limit of Quantitation for details.

Glassware: Class-A digestion tubes should be purchased, or the calibration of the disposable digestion tubes must be verified, to within 3%, for each lot received.

EQUIPMENT (see Appendix_5 for Instrument Conditions)
Leeman Automated Mercury Analyzer, Model Hydra AA
Heating block capable of maintaining a temperature of 90-95°C Calibrated annually along with the thermometers for temperature control within +/- 2C by SOP for Temperature Controls
0.45 µm Syringe Filters, Sartorius
Disposable 70 mL digestion tubes, CPI Catalog # 4370-010010

Note: If the digestion tubes are not certified by the manufacturer or vendor as Class-A, the accuracy of each lot must be verified prior to use according to SOP for Volumetric Labware. This is done by selecting 5 digestion tubes from the lot and filling them to 50mL with 20°C deionized water and weighing it on an analytical (4 decimal) balance; the weights must fall within 3% of the expected weights (48.5 – 51.5g) or the lot may not be used for direct measurement of sample or digestate volume.

PREPARE THE CALIBRATION STANDARDS

Initial calibration standards: Add the following volumes of a 0.100 µg/mL mercury working standard to a series of digestion tubes then follow the digestion procedure below, starting at Step-13 so the volume of the standards matches that of the samples. The LIMS ID of the 100 µg/mL working standard must be documented in the digestion log. These standards expire after 24 hours.

Cal Level	Final Concentration (ug/L)	Volume of 0.100µg/mL Working Std (mL)	Final Volume (mL)
Cal Zero	0.0	0.00	50
Std #1	0.2	0.10	50
Std #2	0.5	0.25	50
Std #3	2.0	1.00	50
Std #4	5.0	2.50	50
Std #5	10.	5.00	50

ICV and CCVs: The Initial Calibration Verification (ICV) standard must be made from intermediates obtained from a different manufacturer than those used to make the initial calibration curve and the Continuing Calibration Verification (CCV) standards may be prepared from either of the sources. *Note:* The US Army Corp of Engineers recommends that the CCV's be prepared from the same source standards as the ICAL curve. An acceptable ICV must be analyzed before any samples are loaded. If the ICV does not pass acceptance criteria and samples were analyzed immediately following it, the entire

calibration must be reanalyzed, as there is no way of determining what affect the sample matrix would have on any subsequent ICV analysis.

An instrument blank should not precede each CCV – if it's necessary, the instrument rinse/bake times must be adjusted instead. Instrument Blanks may be analyzed after high concentration samples regardless of whether a sample or CCV follows (see analysis/sequence section)

Calibration Verification Acceptance Limits: These standards expire after 24 hours.

		Spike Level (ug/L)	Lower Limit (ug/L)	Upper Limit (ug/L)
ICV	(±10%)	5.00	4.50	5.50
CCV1	(±20%)	2.50	2.00	3.00

DIGESTION PROCEDURE

1) Turn on the digestion block and allow it to come to temperature (95°C).

2) Remove samples from the coldroom and allow them to come to room temperature.

3) Calibrate the auto-pipette at each of the volumes needed to spike the batch QC samples and create the initial calibration standards. Document this calibration in the pipette benchbook.

4) Select enough disposable digestion tubes for the calibration standards, samples, and batch QC. Label the tubes with the sample number, standard concentration, or QC number.

5) Review the job sheets to determine if a client requested an MS/MSD on their particular samples. If not, select a sample for batch QC so that matrix QC is rotated throughout the laboratory's clients, so that no one client's samples predominate over a period of time.

6) Prepare the standards as described in Appendix 2 in a series of digestion tubes and digest the standards along with the samples.

7) Line the samples up on the counter or cart in order of increasing job number and increasing sample number; this will help prevent samples from being switched during the aliquotting process.

8) Shake the first sample to ensure homogeneity prior then immediately decant 50mL sample, or an aliquot diluted to 50 mL, to the 70 mL digestion tube labeled with that sample number.

9) Place the digestion tube in the rack and recap the original bottle.

10) Repeat Steps 7 & 8 for the remaining samples, each time checking the bottle label against that on the digestion tube before decanting the sample. Place the digestion tubes in order of increasing job number and sample number.

11) For the method blank, add 50mL of deionized water to the digestion tube.

12) For the blank spike and blank spike duplicate, measure 50mL aliquots of deionized water into the tubes labeled “BS” and “BSD”, then add 1.25 mL of the 0.1 ppm Hg calibration solution to each of the tubes.

13) For the MS and MSD, measure additional 50mL aliquots of the selected sample into the tubes labeled “MS” and “MSD”, then add 1.25 mL of 0.1 ppm-Hg solution to those two tubes.

14) Once all of the samples and batch QC have been measured out, add 2.5 mL concentrated sulfuric acid and 1.25 mL concentrated nitric acid to every digestion tube. Record the manufacturer and lot# of the acids in the digestion benchbook.

15) Add 7.5 mL of 5%-potassium permanganate to each tube and allow the tubes to stand for at least 15 minutes. Record the C&T ID of the reagent in the digestion benchbook.

16) Add 4 mL of 5%-potassium persulfate to each tube. Record the C&T ID of the reagent in the digestion benchbook.

17) Cap & invert to mix.

18) Put the tubes in the block digester.

19) Set the block digester to maintain a temperature of 90-95°C for two hours. In the digestion log, record the time and temperature when the samples are placed into the water bath.

    Note: The temperature readout of the digester will typically be higher than the actual temperature of the digestion tubes, so the actual digestion temperature should be monitored using a thermometer in a reagent blank.

20) While the samples are digesting, before returning the original sample bottles to the coldroom, write the sample numbers and <u>bottle letters</u> in the benchbook.

    Write “MSS” in the ‘Comments’ field next to the sample used for the matrix QC, to indicate that it is the “Matrix Spike Sample”.

21) Record the standard IDs, reagent Ids, and digestion start time in the benchbook.

22) Return the original sample bottles to the coldroom and sign them back into the log.

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23) After the 2 hour digestion cycle is complete, remove the digestates from the water bath and allow them to cool to room temperature. In the benchbook, record the time the digestates were removed from the water bath.

24) Add 3 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate. Add an excess of reductant in 3 mL increments until permanganate is reduced completely (purple color fades to colorless). Do this step at least 30 seconds before the addition of the stannous chloride solution.

    Record the C&T ID of the reagent in the prep log.

    Note: If one sample requires additional reductant, the same volume of reagent must be added to all samples, batch QC, and standards. This ensures that the final volume of the calibration standards matches that of the samples.

25) If any of the digestates contain sediment or precipitate, filter the samples and all batch QC through 0.45 µm syringe filters, into labeled autosampler tubes.

    If the samples do not contain sediment or precipitate, simply decant the samples into labeled autosampler tubes.

26) Before continuing, complete the digestion benchbook record and enter the prep information in LIMS. Complete the Prep section of the Data Review Checklist.

27) Print the LIMS prep entry report and have the Department Manager or a qualified analyst review and sign the benchbook and prep entry before analyzing the digests.

INSTRUMENT CALIBRATION

1) While the samples are digesting, refill the stannous chloride reservoir and make sure the inlet tube is submerged in the reagent; document the reagent ID in the digestion log. Turn on the lamp and allow the instrument to equilibrate for about an hour.

1.) After digestion, load the calibration standards onto the autosampler tray in order of increasing concentration.

2.) Add instrument blanks before and after the ICAL standards to demonstrate that the low-level standard was not influenced by instrument contamination and that analytes at the high-level concentrations will not carryover into real-world samples.

3.) Verify that the correlation coefficient (r) is $\geq$0.995. If one level of the initial calibration curve has a particularly deviant response factor, that level may be rerun so long as it immediately follows the last calibration standard and no sample digests have been injected.

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4.) Load the ICV after the instrument blank that follows the calibration standards. The ICV, prepared from standards obtained from a second manufacturer, must be analyzed to verify that the standards used to create the initial calibration curve were prepared correctly.

5.) Before loading samples, verify that the ICV recovery is within the 90-110% acceptance limits and that no mercury was detected in the instrument blank. If the ICV is outside the acceptance limits, it may be reanalyzed once, so long as no sample digests have been injected (which could change the instrument response).

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Calibration Verification (ICV & CCV) Acceptance Limits

		Spike Level (ug/L)	Lower Limit (ug/L)	Upper Limit (ug/L)
ICV	(± 10%)	5.00	4.50	5.50
CCV2	(± 20%)	5.00	4.00	6.00

SAMPLE ANALYSIS

6.) After the instrument has been calibrated, set up the remainder of the sequence as outlined below, with a CCV/CCB pair analyzed after every 10 samples (at minimum), including QC, and at the end of the sequence:

Initial Calibration Standards
Initial Calibration Verification (ICV, second source) standard
Initial Calibration Blank (ICB)
Prep Blank
Blank Spike
Blank Spike Duplicate
Sample
Matrix Spike
Matrix Spike Duplicate
4 more samples
Continuing Calibration Verification (CCV) standard
Continuing Calibration Blank (ICB)
10 samples
CCV
CCB
etc..

The CCV concentration must be alternated across the calibration range (the high and low ICAL points should not be used as CCV's) over the course of the sequence.

7.) Place the samples in an autosampler rack and program the autosampler appropriately. The instrument will automatically add the 10% stannous chloride reagent and analyze the samples. See Appendix_7 for instrument procedures.

8.) **Dilutions:** If the sample concentration is greater than the highest point in the calibration curve, and the digest was completed within the past 24 hours, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range, using Appendix_3 as a guide.

If the digestion was completed more than 24 hours previously, redigest the sample in a new batch and dilute the digestate prior to analysis.

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9.) If a **Serial Dilution (SER)** is needed, measure 2.0 mL of the MSS (unspiked aliquot of the "matrix spike sample") into a digestion tube. Add 8.0mL of the digested blank (to match the reagent concentrations) and swirl to mix. Decant into an autosampler tube and add to the sequence.

10.) If a **Post-Digestion Spike (PDS)** is needed, pipette 10.0 mL of the MSS into a digestion tube. Add 0.5 mL of a 0.1 mg/L standard. Cap and swirl to mix. Decant into an autosampler tube and add to the sequence.

DATA REVIEW & REPORTING

After the sequence is complete, review the data to determine if the data may be reported or if reanalysis or redigestion is necessary. Any corrective action involving reanalysis of the digests must occur within 24 hours of the digestion, or the digestion must be repeated.

Quantitation is based on comparison of the measured absorbance for the sample against the initial calibration curve, with adjustments for the sample preparation concentration factor and instrument dilution factor. See Appendix 4 for example calculations. Concentrations are expressed as micrograms per liter (µg/L) or milligrams per kilogram (mg/kg).

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

- Examine the **Continuing Calibration Verification (CCV)** data. The recovery for each should be within 90-110% recovery; if any of the recoveries are outside acceptance limits, use the following to determine the appropriate corrective action:

  a.) If a low recovery is observed, any samples bracketed by the low standard must be reanalyzed.

  b.) If a high recovery is observed and no mercury was detected in the samples, those samples may be reported without reanalysis.

  c.) If a high recovery was observed and mercury was detected in the samples, those samples must be reanalyzed.

- Evaluate the **Continuing Calibration Blank (CCB)** data. Mercury should not be detected in the calibration blanks at any level greater than the method detection limit however, if it is detected, use the following to determine the course of action:

  a.) If mercury is detected above the MDL, but below ½ RL, in the CCBs, narrate the problem on the data review checklist and report the data without reanalysis.

b.) If mercury is detected above ½ the RL but mercury was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c.) If mercury was detected above ½ the RL and mercury was also detected in the sample(s), but the level in the sample(s) is greater than 10x that in the blank, the results may be reported without reanalysis, as the contamination level is considered insignificant in relation to the sample levels.

d.) If mercury was detected above ½ the RL and mercury was also detected in the sample(s) at levels < 10x that in the blank, the samples must be reanalyzed.

- Evaluate the **Method Blank (MB)**. The results for the method (preparation) blank should be < 1/2 RL. If reanalysis confirms the contamination, use the following steps to determine if the sample results may be reported:

  a.) If the concentration in the blank is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

  b.) If mercury was found in the blank but was not detected in the associated samples, the data may be reported and the problem narrated.

  c.) If mercury was found the method blank and was also detected in the associated samples, but the level in the samples is greater than 10x the level in the blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

  d.) If mercury was detected in the blank were also detected in the associated samples, but at levels less than 10x the level in the blank, and reanalysis confirms the problem, the samples containing the contaminant must be redigested and reanalyzed. Initiate a Corrective Action Report (CAR) and redigest and reanalyze all of the affected

- Evaluate the **BS/BSD** data. Both recoveries and the RPD should be within acceptance limits. If either recovery or the RPD fails, immediately reanalyze the failing batch QC sample. If the failure is repeated, use the following to determine the appropriate corrective action:

  a.) If a low recovery is observed, all samples in the batch must be redigested and reanalyzed.

  b.) If a high recovery is observed and no mercury was detected in the samples, those samples may be reported without reanalysis.

c.) If a high recovery was observed and mercury was detected in the samples, those samples must be redigested.

d.) If a high RPD was observed but both recoveries are within limits and no mercury was detected in the associated samples, the samples may be reported without reanalysis.

e.) If a high RPD was observed and mercury was detected in the associated samples, the samples must be redigested.

- **Matrix QC (SSPIKE/SDUP or MS/MSD):** The recoveries and RPD should fall within C&T in-house limits, or the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine any necessary corrective action:

  a.) If the concentration in the sample is greater than the linear range and the sample needs to be rerun for quantitation, report the MS/MSD with a LIMS-flag of ">LR" on those recoveries, without reanalysis.

  b.) If the concentration in the sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a ">LR" flag to those recoveries. Report the data without reanalysis.

  c.) If the concentration is greater than 4x the spiking level, LIMS will apply a "NM" (for "Not Meaningful") flag to those recoveries. Report the data without reanalysis.

  Note: For USACE projects, if the concentration is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

  d.) If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

  e.) If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

- Examine the **sample** data. If the concentration in any extract exceeds that of the most concentrated standard, the results will be flagged '>LR' by LIMS; the extract must be diluted and reanalyzed to bring the response into the calibration range. Diluted sample concentrations must be above the low-CCV level and preferably in the upper half of the calibration range.

  If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

- **Assemble the Data Package**
  1. After all samples and necessary dilutions have been analyzed, print the final report forms on “page 2” letter head paper.

  2. Review these forms to make sure that the correct results were reported and that there are no elements marked “N/A” (results “not available”).

  3. Verify that the prep sheets are complete and signed, that all necessary sample information is present (see section below for details), then complete and sign the “Data Review Checklist”.

  4. Submit the data package to the Department Manager, qualified analyst, or a QC Chemist for second-party review.

  Any changes made by the second-party reviewer must be individually initialed and dated by the reviewer. The second party reviewer must initial and date each user report, make any additional comments on the case narrative and initial and date the completed checklist.

DOCUMENTATION REQUIREMENTS

a.) **Sample Prep Documentation**

A copy of the digestion benchbook page for the sample digestion must be scanned so prep info can be included with reported data. The digestion benchbook entries should include:

- C&T sample ID's and unique container identifier,
- date of sample digestion, initial volume or weight of sample, and final digestate volume,
- time the samples were placed in the water bath and time they were removed,
- identity of QC samples (spikes, duplicates & LCS),
- amount of spikes added and LIMS identification numbers of all spiking solutions,
- a list of all reagents used (C&T ID or manufacturer and lot number),
- indication of whether or not the digests were filtered after digestion,
- any unusual occurrences observed during the digestion procedure

b.) **“Paperless” Package** Procedure:

1. Scan the digestion benchbook page into the scanner. Select Lab > Batch Prep Sheet > Enter Batch Number. Scan.

2. Sign the Batch using the Review App. Confirm the batch prep sheet appears correctly.

3. Sign the Calibration for all instruments and runs for that batch. This is located under the ICAL section on the left side of the Review App beneath the batch number.

4. Sign the sequences in which the jobs were run. This is verifying that the samples were loaded in the correct order and at the correct dilutions as indicated by the sample files in the

software.

5. Sign all the jobs within the batch. Verify that the reports have been printed and that all the runs are correctly chosen. Insure that there are no samples marked as "NA" due to missing Mercury runs. Samples may be marked "NA", however, due to other runs needed within the department.

6. The data package will be reviewed again by the Department Manager.

POLLUTION PREVENTION
Vent the mercury analyzers into the fume hood. Purchase only sufficient standard and reagent volume to use within the expiration date, to reduce the volume of laboratory waste.

WASTE DISPOSAL
Excess sample volume must be returned to the coldroom for later inclusion in the lab's aqueous waste stream. The shelf-life for mercury digests is only 24 hours, so the digests may be discarded upon completion of the analysis, after the sequence has been reviewed and no further dilutions or reanalysis is needed. Excess digestate volume and instrument waste should be included in the 'Corrosives' waste stream.

REVISION HISTORY
This is revision 16, revision 15 was reviewed and the following changes were made:

- Removed: alternating CCVs
- Changed spike level
- Removed and added instruments

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APPENDIX_1: **MERCURY WATER DIGESTION SUMMARY**

Sample Volume:	50 mL
Spike:	1.25 mL of HG 0.1 STD
1st Digestion:	+ 2.5 mL concentrated H_2SO_4 + 1.25 mL concentrated HNO_3 7.5 mL $KMnO_4$ solution Rest at room temperature $\geq$ 15 minutes
2nd Digestion:	+ 4 mL 5% Potassium Persulfate 90 - 95 °C 2 hours (add $KMnO_4$ crystals if color fades)
Final:	+ 3.0 mL NaCl-Hydroxylamine HCl Repeat until appearance is stable & colorless Bring to 50 mL with DI water
Filtration:	0.45 µm Syringe Filter, including all batch QC

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APPENDIX _ 2: REAGENTS & STANDARDS

The standards and reagents listed below are those in use at the time this procedure was written. Alternate supplies may be used so long as the resulting mix will contain all of the target compounds, are of equivalent quality and meet all other calibration, quality control, and traceability requirements.

REAGENTS

Document the preparation of all reagents, including dilutions into DI water, in the reagent prep benchbook. Assign a unique ID to each reagent prepared, based on the manufacturer and the date prepared. Record this ID in the digestion benchbook each time the reagent is used.

Label each reagent with the reagent ID, concentration, prep date, and expiration date. Expired, discolored, or contaminated reagents should be discarded and the bottle cleaned before reuse.

Deionized Water, ASTM Type II (ASTM D1193)

Hydroxylamine Hydrochloride ($H_2NOH.HCl$), reagent grade
Fisher Scientific catalog # H330-500
Store in a tightly sealed plastic bottle at room temperature for up to 5 years

Nitric Acid (HNO_3), concentrated, InstraAnalyzed grade
JT Baker catalog # 7697-37-2
Store in a Corrosives cabinet for up to 2 years

5% Nitric Acid (HNO_3)
Add 50mL concentrated HNO_3 to 950mL deionized water.
Store at room temperature for up to 1 year

Potassium permanganate ($KMnO_4$), reagent grade, mercury free
Fisher Scientific catalog # P279-212
Store in an amber glass bottle at room temperature for up to 5 years

Potassium permanganate, mercury-free, 5% (saturated) solution (w/v):
Dissolve 50 g of potassium permanganate in 1,000 mL of deionized water.
Store in an amber glass bottle for up to 3 months

Potassium persulfate ($K_2S_2O_8$), reagent grade
Fisher Scientific catalog # P281-500
Store at room temperature for up to 5 years

Potassium persulfate, 5% solution (w/v):
Dissolve 50 g of potassium persulfate in 1,000 mL of deionized water.
Store in an amber glass bottle for up to 1 year

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Sodium Chloride (NaCl), reagent grade
Fisher Scientific catalog # S671-500
Store in a tightly sealed plastic bottle at room temperature for up to 5 years

Sodium chloride-hydroxylamine hydrochloride solution:
Dissolve 120g of sodium chloride and 120g of hydroxylamine hydrochloride in deionized water and dilute to 1,000 mL. Store in a glass bottle for up to 3 months

Stannous chloride ('flow reagent'):
Add 40g stannous chloride and 30 mL HCl to 200 mL of deionized water and then bring to a total volume of 400 mL. This mixture may require agitation to dissolve residual tin chloride. Store reagent in a plastic bottle. Prepare weekly or more frequently if the solution becomes discolored.

Sulfuric Acid Concentrated (H_2SO_4), concentrated, InstraAnalyzed grade
JT Baker, VWR catalog # JT9691-3
Store in a Corrosives cabinet for up to 2 years

Source Standards

Source standards are those purchased from a chemical manufacturer or vendor. For source standards, the LIMS S-name is unique to both the composition (compound list) of the standard *and* to the vendor of that standard. A new S-name must be assigned whenever the composition is changed or when the standard is obtained from a different vendor; the information must then be entered in the "Standard Definitions" table before the new standard is assigned an S#.

Certificates of Analysis should be obtained from the vendor of each source standard; each standard should be traceable to NIST. If the supplier did not provide a certificate, call and request that a copy be faxed. Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is one year from date received.

Enter the lot#, date received, and expiration date of each source standard into LIMS immediately upon receipt, using the Standards Menu "Standard Inventory".

Label the Certificate of Analysis with the LIMS S#, date received and expiration date (if not already listed on vial) then file the certificates the 3-ring binder. Label each vial with the contents, LIMS S#, and expiration date. Store each source standard in the vendor's bottle at room temperature, until the assigned expiration date.

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Analytes	Concentration	Supplier & Catalog#	LIMS S-Name
Mercury, primary source std. Plasma-Pure Part # 603-8062	1,000 ug/mL	Leeman # 7439-97-6	HG LEEMAN
Mercury, second source std.	1,000 ug/mL	CPI # S4400-1000331	HG SS

Working Standards

Working standards are those prepared at C&T. For working standards, the LIMS S-name is not unique to the source standard vendor but *is* unique to the compound list and concentrations contained in the working standard; if the concentration or compounds in the working standard changes, a new S-name, compound list and concentrations must be entered in the "Standard Definitions" table before the standard is logged in and assigned an S#. It is very important to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

Verify that the LIMS expiration date does not exceed the Source Standard expiration date. If it does, change the working standard expiration date to that of the source standard. **The expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**.

In the Standards Benchbook, enter the prep date, LIMS S#, concentration, and volume of each source standard used, the LIMS S-name, final volume and concentration of the working standard, expiration date, and prep chemist's initials.

In LIMS, enter the prep chemist's initials, prep date, and S# of all source standards used to make the working standard; LIMS will then assign a working standard number (S#).

Prepare working standards by diluting source standards to volume in a Class-A volumetric flask. Label the standard vial with the contents, the LIMS S#, the expiration date of the standard, and the prep chemist's initials.

Procedure:

1.) Prepare all working standards in a **200 mL Class-A volumetric flask**, initially adding about 200mL DI water.
2.) Add 10mL concentrated nitric acid to about 100mL DI water (so the final acid concentration in 200mL final volume will be 5%). Swirl to mix.
3.) Add the volumes and standards listed below to make each standard.
4.) Bring to volume with DI water.
5.) Cap and invert three times to mix. Allow sufficient time, during each inversion, to mix thoroughly.

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All volumes given below may be adjusted proportionally to yield more or less of the solution required. All spiking standards should be analyzed to verify their concentration **PRIOR TO USE**. The data from these screens should be stored in the appropriate binder.

MERCURY STANDARDS PREP

Working Standard & Conc.	WS Conc. (ug/mL)	Using "Source" Std	Add Vol (mL) Source Std	LIMS S-Name	Shelf Life
Primary Source Intermediate	10.0	HG LEEMAN	2.0	HG 10 STD	3 mo
Primary Source Working Std	0.100	HG 10 STD	2.0	HG 0.1 STD	24 hr
2nd Source Intermediate	10.0	HG SS	2.0	HG 10 REF	3 mo
2nd Source Working Std.	0.100	HG 10 REF	2.0	HG 0.1 REF	24 hr

DAILY CALIBRATION STANDARDS

CAL Standard & Conc. (µg/mL)	Final Volume (mL)	Using Source Std	Add Vol (mL) Source Std	LIMS S-Name
CAL-Zero 0.0 ug/L	50	DI water	0.00	
CAL-1 0.2 ug/L	50	HG 0.1 STD	0.10	
CAL-2 0.5 ug/L	50	HG 0.1 STD	0.25	
CAL-3 2.0 ug/L	50	HG 0.1 STD	1.00	
CAL-4 5.0 ug/L	50	HG 0.1 STD	2.50	
CAL-5 10.0 ug/L	50	HG 0.1 STD	5.00	
ICV 5.0 ug/L	50	HG 0.1 REF	2.50	HG ICV
CCV-1 0.5 ug/L	50	HG 0.1 STD or REF	0.25	CCV1 HG
CCV-1 2.0 ug/L	50	HG 0.1 STD or REF	1.00	CCV2 HG
CCV-1 5.0 ug/L	50	HG 0.1 STD of REF	2.50	CCV3 HG

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APPENDIX _ 3: SAMPLE DILUTIONS

If the sample concentration is greater than the highest point in the calibration curve, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range. This dilution must be injected onto the instrument within 24 hours of completion of the digestion; if the digestion was completed more than 24 hours previously, redigest the sample in a new batch and dilute the digestate prior to analysis.

Examine the original sample data to determine what dilution factor will be required to bring the absorbance down to the middle of the calibration range.

Using calibrated auto-pipettes, add the following volumes sample followed by HNO_3 acidified (5% Vol:Vol) deionized water to a 15mL disposable centrifuge tube. Cap and invert three times to mix, allowing sufficient time for complete mixing with each inversion.

DILUTIONS

Instrument Dilution Factor (IDF)	Digestate Volume	Add Volume (mL) Acidified DI Water	Final Volume (mL)
2	5.0 mL	5.0	10
5	2.0 mL	8.0	10
10	1.0 mL	9.0	10
20	0.50 mL	9.5	10
50	0.20 mL	9.8	10
100	0.10 mL	9.9	10

If a sample should need a dilution of more than 100x, prepare a 100x dilution first, then use that to make subsequent dilutions.

SERIAL DILUTIONS

Instrument Dilution Factor	Using Primary Dilution	Volume of Primary (100x) Dilution	Add Volume (mL) Acidified DI Water	Final Volume (mL)
500	100x	2.0 mL	8.0	10
1,000	100x	1.0 mL	9.0	10
2,000	100x	0.5 mL	9.5	10
5,000	100x	0.2 mL	9.8	10
10,000	100x	0.1 mL	9.9	10

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APPENDIX _ 4: CALCULATIONS

SAMPLE CONCENTRATION

Raw Amt (ug/L) = (m * intensity) + b

Where: m = first-order coefficient (slope) from initial calibration (LIMS a1)
b = intercept (LIMS a0)

LIMS then applies the sample prep information to calculate the mercury concentration in the sample by dividing the total ug by the prep dilution factor.

Conc (ug/L or mg/Kg) = Raw Amt * Vf / Si * IDF

Where: Vf = Final Digestate Volume (mL)
Si = Sample Initial Volume (mL) or Weight (g)
IDF = Instrument Dilution Factor

BATCH QC CALCULATIONS

Percent Recovery (%R):
The recovery is the measured concentration divided by the true concentration of the spike.

%Recovery = (Cf – Cs) / (Cws * Vws/ S) *100

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard
S = Sample weight or volume

Relative Percent Difference (RPD):
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

%RPD = |(Cs - Cdup)| / ((Cs + Cdup)/2) * 100

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

For soil MS/MSDs where the sample weights are not weight-targeted, the expected concentrations will vary with sample weight (because the same volume of spike standard is being added to different weights of sample) and must be accounted for when calculating RPD:

$$\%RPD = |((Wms/Wmsd)*Cms - Cmsd)| / (((Wms/Wmsd) * Cms + Cmsd)/2) * 100$$

CALIBRATION EQUATIONS

%Drift used calibration verification
Used for calibration verification standards, to determine if the instrument is still in control:

$$\% \text{ Difference (\% Drift)} = \%D = ((C1 - C2) / C1) * 100$$

Where: C1 = Concentration of the Calibration Verification Standard
C2 = Measured concentration

Linear Regression Equations

$$Y = mx + b$$

Where: y = response (Ax for external standard, or Ax/Ais for internal standard)
Where Ax = Area of compound
Ax / Ais = Area of compound divided by area of internal std
x = concentration (Cx for external standard, or Cx/Cis for internal standard)
m = slope
b = intercept

$$\text{Slope (m)} = [(\sum wx_iy_i * \sum w) - (\sum wx_i * \sum wy_i)] / [(\sum w * \sum wx_i^2) - (\sum wx_i * \sum wx_i)]$$

$$\text{Intercept (b)} = y_{avg} - (m * x_{avg})$$

Correlation Coefficient (r)

$$r = \frac{[(\sum w * \sum wx_iy_i) - (\sum wx_i * \sum wy_i)]}{\sqrt{\{[(\sum w * \sum wx_i^2) - (\sum wx * \sum wx_i)] * [(\sum w * \sum wy_i^2) - (\sum wy_i * \sum wy_i)]\}}}$$

$$\text{Coefficient of Determination } (r^2) = r * r$$

Where: x_i = individual values for the independent variable (concentration)
y_i = individual values for the dependent variable (response, area)
w = weighting factor (for no weighting w = 1)
x_{avg} = average of the x-values
y_{avg} = average of the x-values

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STANDARDS PREP & OTHER QC

Spiking Batch QC for Waters
Calculate the volume of working standard to add to make the LCS/ MS/ MSD using the following:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where: Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/L)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Volume of Sample (mL)

Spiking Batch QC for Soil
Calculate the volume of working standard to add to make the LCS/ MS/ MSD using the following:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where: Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/Kg)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Mass of Sample (g)

Make a Working Standard from a Source (Stock) Standard:
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where: Vss = Volume of Source Standard (mL) needed to make new standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

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APPENDIX_5: INSTRUMENT CONDITIONS

The conditions listed below were those in use at the time this document was written and may be modified at the analyst's discretion, so long as those changes are documented in the instrument maintenance log.

Instrument: MET-14, Leeman Hydra AA
MET-34, Leeman Hydra AA II

Argon	Purity	99.99%
	Max Pressure	60 psi
	Flow Rate	0.6 L/min
Oven Radiator Temperature:		125 °C (to maintain 50°C gas temperature)
Lamp Current		6 – 10 mAmps
Auto Sampler		30s sip duration 75s rinse time
Peristaltic Pump Rate		6.0 mL/min Sample 0.8 mL/min Reagent (Stannous Chloride)
Auto-Integration		4 replicates
Baseline Correction		1 point

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APPENDIX_6: INSTRUMENT MAINTENANCE

All parts & supplies can be ordered from LEEMAN; see instrument manual for part numbers. Alternate suppliers and parts may be used at the analyst's discretion.

All maintenance events must be documented in the instrument maintenance benchbook. The benchbook entries should include the following:

- reason for the maintenance,
- the initials of the person performing the maintenance and the date performed,
- description of maintenance performed,
- resolution (If the maintenance was due to poor instrument performance, did the maintenance solve the problem?)

Pump windings must be replaced whenever cracked or leaking.

The **drying tube** (gas-liquid separator) should be cleaned by sonicating in 1:1 nitric acid/ DI water for 30 minutes whenever the glass begins to turn yellow from tin chloride precipitation.

The **gas-liquid separator** should be cleaned with spectrophotometric grade isopropyl alcohol if the cell appears smudged or dirty.

The **lamp** should be ordered when the voltage is at 10mV and replaced when the voltage reaches 15mV.

DIGESTION BLOCK INSTRUCTIONS

1) Turn on the block.
2) On the keypad, press ENTER/ START.
3) Select AUTO.
4) Set the temperature to 95°C.
5) Allow block to warm up for 20 - 30 minutes.
6) Use a thermometer, in a water-filled digestion tube, to verify that the temperature is 95°C.
7) Adjust the temperature setting as needed so that the digestate temperature will be maintained at 95°C.
8) Place the digestion tubes in the bath.
9) Set the timer for 30 minutes for soil samples (2 hours for water).
10) Document the time and temperature in the digestion log.

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APPENDIX_7: USING THE LEEMAN SOFTWARE

Install # 62541
800-533-6267 David Sherwin, Field Service Engineer

The Hg analyzer provides a windows interface that interacts with LIMS once a batch has been entered, prepped and assigned a batch number. Before using the windows interface you must have a LIMS batch number and LIMS prep information.

1. Open the WinHg Runner Program.

   1.1. Click on the icon in the quick launch bar
   1.2. Open the program from Start->Programs->WinHg->WinHg
   1.3. Execute the program file located at C:\Hg\WinHg.exe
   1.4. If all else fails, you can click Start->Run and on command line type "C:\Hg\WinHg.exe"

2. Use the functions under the **Control** tab to control the parts of the instruments such as the pump, gas and lamp. When starting up, go to this tab first. Here you will:

   2.1. Turn on the lamp
   2.2. Turn on the pump (5mL/min)
   2.3. Turn on the gas (0.6 L/min)

   Note: Should you ever get very negative calibrations (~ -2000 or more), use the lamp adjust feature on this tab.

3. Create a new dataset (sequence) by selecting "New Dataset" from File menu.

   3.1. A window will pop up asking for new dataset name.
   3.2. Enter the date in MMDDYY format, e.g. 041305.
   Note: For the second batch to be run on any given day, you will need to append a letter to the name, for example, 041305a, 041305b and so on.
   3.3. Another window will pop-up asking for batch number.
   3.4. Enter your current batch number here.

4. Use the **Standards** Tab to set up the calibration once your standards are made and in the autosampler tray.

   4.1. On left hand side, depress the first six buttons (S1-S6), corresponding to your standards (0.0, 0.2, 0.5, 2.0, 5.0, 10.0),
   4.2. Depress the "Rep 1" button, and click "Stnd Auto".
   4.3. This will begin to establish your calibration curve.
   4.4. Click on the Database (DB) button on the top tool bar.
   4.5. Select the "Cal Curve" Tab to view your calibration curve in progress.

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4.6. If your calibration curve has a Rho factor (correlation coefficient) of ≥0.995, click on the "Accept" button on the "Cal Curve" tab of the DB application.

4.7. Print the calibration curve by either:
   a. clicking the print button from the top toolbar of DB application, or
   b. selecting Print from the File menu

4.8. Delete any calibration points not included in the ACCEPTED curve, by selecting the line and clicking on the delete button of the Report tab. *Note:* You can also uncheck the box to the left of the sample ID; however, this change is temporary, as the box will automatically be re-checked as additional samples are run and the list is "refreshed"

5. Use the **Rack Editor** interface to enter information about the samples in the autosampler rack.

6. Use the **Reports** to review the sequence and select the records to submit to LIMS.

6.1. Carefully review the samples in the sequence (or "Record List" as it is referred to on the Report tab of the DB application). Be sure that only the calibration points used in the accepted curve are included in this list. These will be the six StdXRepY that precede the ICV. *Note:* Whenever you reset calibration data (from the standards tab of the Runner application), the curve is reset, but the readings still exist.

6.2. Uncheck any samples you wish to omit.

6.3. Generate the reports by clicking on the "Generate Report" button of the Report Tab.

6.4. Format= Report, Destination = Printer.

6.5. Review this printed report carefully before submitting the data to LIMS. The data that appears on the printout also appears on the **Viewer** Tab of the DB application.

6.6. Format = PRN, Destination= Disk File
Note: The path should be G:\limsinp\met04\batchnumber.PRN.
To set the destination, you must navigate to the .../met04 directory by
   1.5.1 Clicking on the button to the right of the output file path.
   1.5.2 A dialog box will pop-up; in that folder, save the file as its batch number.
   1.5.3 Click Save, or press <Enter>, then Click on "Generate Report".

6.7. After this step is complete, the data is dealt with entirely through the LIMS system.

Note: The default display path is "C:\Hg". This is the local hard-drive directory where the startup.ini, report specifications, protocol parameters and other instrument/system files are housed. Be sure that the "Report Specs" pull down menu displays "DLNEW". This is the format file that enables your instrument to talk to the Laboratory Information Management System (LIMS).

TROUBLE-SHOOTING LEEMAN HYDRA DATA

From the LEEMAN HYDRA AA User's Manual
The WinHG main program has seven tabs that outline its basic functions: main, control, standard, sample, output, report and utility. You will use all of these tabs on a daily basis, except for main and utility.

The typical operation of the HYDRA AA follows a sequence as below:

- Load or create a protocol which defines the operating parameters of the system
- Load or create a dataset where the data will be stored.
- Turn on Gas, Pump, and Hg lamp
- Sample rack editing (Add sample names, Dilution factors, Weight factors, etc.)
- Standard concentration and unit entry.
- Check standard concentration and limits entry.
- Run standards for calibration of HYDRA AA
- Accept calibration curve
- Run samples using calibration stored in protocol.
- Post run reporting of data.

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Curtis & Tompkins, Ltd

TOTAL EXTRACTABLE HYDROCARBONS

'TEH' by GC/FID Method EPA 8015B and 8015D

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Curtis & Tompkins, Ltd

"TEH" by GC/FID Method EPA 8015B and 8015D

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TOTAL EXTRACTABLE HYDROCARBONS

"TEH" by GC/FID Method EPA 8015B and 8015D

SCOPE

This procedure details the identification and quantitation of Total Extractable Hydrocarbons (TEH) in the dichloromethane extracts. This procedure uses a gas chromatograph (GC) to separate the components followed by a Flame Ionization Detector (FID) that detects the components as they pass through the detector. This method is applicable to the determination of jet fuel, kerosene, diesel, motor oil, or other hydrocarbons between the carbon-markers C-10 to C-50.

Reporting limits for water samples are 50 µg/L for jet fuel, kerosene, and diesel and 300 µg/L for motor oil, transformer oil, and Bunker C. Reporting limits for soil samples are 1 mg/Kg for jet fuel, kerosene, and diesel and 5 mg/Kg for motor oil, transformer oil, and Bunker C.

REFERENCES

Analysis Methods:
EPA 8015B, *Non-Halogenated Organics using GC/FID*, SW-846, December 1996
EPA 8015C, *Non-Halogenated Organics using GC/FID*, SW-846, Nov 2000
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Tri-Regional Recommendations, Revised 10 August 1990
8015AZ, "C6-C32 Hydrocarbons in Soil", Arizona Dept of Envir Quality, Rev.1, 9/25/98

Extraction/Preparation Methods:
EPA 3500B, Organic Extraction & Sample Preparation, SW-846 Update 3, Dec 1996
EPA 3500C, Organic Extraction & Sample Preparation, SW-846 Feb 2007
EPA 3510C, Separatory Funnel Liquid-Liquid Extraction, SW-846 Update 3, Dec 1996
EPA 3520C, Continuous Liquid-Liquid Extraction, SW-846 Update 3, Dec 1996
EPA 3535, Solid-Phase Extraction, SW-846 Update 3, Dec 1996
EPA 3535A, Solid-Phase Extraction, SW-846 Feb 2007
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EPA 3545A, Pressurized Fluid Extraction, SW-846, Feb 2007
EPA 3550B, Ultrasonic Extraction, SW-846 Update 3, Dec 1996
EPA 3550C, Ultrasonic Extraction, SW-846, rev 3 Feb 2007
EPA 3580A, Waste Dilution, SW-846 Update 1, July 1992
California Leaking Underground Fuel Tank Field Manual (LUFT Manual), October 1989
Tri-Regional Recommendations, Revised 10 August 1990

Cleanup Methods:
EPA 3630C, Silica Gel Cleanup, SW-846 Update 3, Dec 1996

Related C&T Procedures & Other Guidance Documents:
SVOC 5.1.1, 'TEH QC Acceptance Limits, Table-1'

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QA 1.4, Balance Calibration Check & Maintenance
QA 1.5, Calibrating & Maintaining Temperature Controls
QA 1.6, Pipet Calibration Check Procedures
QA 4.1, Establishing Control Limits
QA 4.4, Determining Method Detection Limits (MDL)
NELAC Chapter 5, Quality Systems, June 2003
DoD Quality Systems Manual, Version 4.1, April 2009
Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Rev 4.2 October 2010

PRESERVATION & HOLDING TIME

Preservation:	No chemical preservation. Store samples and extracts at 4° C.	
Holding time:	California/LUFT H_2O:	14 days from collection to extraction.
	Other H_2O:	7 days from collection to extraction.
	Soil:	14 days from collection to extraction.
	Extract:	40 days after extraction.

Note: For Arizona, soil samples must be analyzed within 14 days of sample collection.

QC REQUIREMENTS

Mid-range continuing calibration verification (CCV) standards of diesel (and other hydrocarbons if requested) must be analyzed prior to the analysis of samples to verify the calibration curve. Additional CCV standards should be analyzed after every 10 samples “a bracket”, not including batch QC, and at the end of the sequence. Alkane Retention-Time Verification (carbon-marker) standards must be analyzed every twelve hours or every other bracket.

The %D for the fuel CCV’s must be $\leq$ 15%. If one of the fuel standards does not meet this requirement, any extracts that are being analyzed for this fuel and were analyzed after the last passing CCV must be reanalyzed. The carbon-markers must fall within the expected retention time for those carbon ranges; if the RT’s drift outside the RT windows, the method must be updated and all associated data reprocessed against the corrected retention times. The method must be updated and the data will only be re-analyzed when it’s impacted by the retention shift.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM) and the method 8015D requirement for the ICV & CCV %D is $\leq$ 20%.

Two surrogate compounds are added to each CCV standard, sample, method blank, and spike to monitor the performance of the analytical system. A method blank (BLANK), blank spike (BS) and blank spike duplicate (BSD), or lab control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) will be analyzed for each batch of 20 samples or less. For projects or sites that are known or suspected to contain high levels of hydrocarbons or matrix interferences, a single sample spike (SSPIKE) and sample duplicate (SDUP) may replace the MS/MSD.

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Acceptance limits for the surrogates and spikes are updated semiannually, based on control charts of the previous year's results. See the associated SOP *TPH-E Laboratory Control Limits; Table-1* for current in-house QC acceptance limits.

Method Detection Limit (MDL) studies must be performed annually, at minimum, on each instrument and matrix. See the QA SOP 4.4 "Determining Method Detection Limits" for details.

Limit of Detection (LOD), must be determined quarterly and is based on the analysis of a laboratory control sample (MDL verification standard) that is spiked 1 to 4 times the MDL. See the QA SOP for Establishing and Validating Method Detection Limits or Limits of Detection for details.

Limit of Quantitation (LOQ) must be determined quarterly and is based on a laboratory control sample that is spiked 1 to 2 times the reporting limit. It is only analyzed once per method. See the QA SOP for Establishing and Verifying the Limit of Quantitation for details.

SAFETY

Assume that all extracts contain potentially toxic materials and should be handled with care. The chromatograph's injection port and detectors are hot and should be handled with caution. Take appropriate precautions when handling pressurized gas cylinders; all gas cylinders should be chained to a wall or benchtop when in use.

EQUIPMENT

Gas chromatograph:	Hewlett-Packard 5890 with HP-7673 Autosampler Agilent 6890N with Agilent G2614A (7683B) Autosampler Agilent 7890A with Agilent 7683B Autosampler
Guard column:	5 meter x 0.32mm I.D.
Guard column:	5 meter x 0.32mm I.D. IP Deactivated (Restek, Catalog # 10044)
GC column:	15 meter x 0.32mm I.D., 0.1 um df, 100% polysiloxane (Restek Rtx-1, Catalog # 13321) RX -1ms
Detector:	Flame ionization detector (FID)
Chromatography Data System:	Agilent EZ Chrom, Version 3.1.7

Alternate supplies and vendors may be used so long as all QC requirements are met.

INTERFERENCES

The purpose of this analysis is primarily to determine the identity and concentration of petroleum fuels; however, non-petroleum hydrocarbons may also be detected. These are called "biogenic" interferences and may consist of vegetable oils, fatty acids, sugars or other polar analytes. This type of interference is typically seen in aquatic water or sediment samples, due to algae or other marine life. Also, surface soils at agricultural sites where decomposing vegetable or animal matter may contribute high levels of non-petroleum hydrocarbons. Because petroleum products are not very polar, a silica gel cleanup will remove the polar biogenic material without affecting the non-polar petroleum products.

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MAINTENANCE

Maintenance is performed when needed. Remove the 'solvent' vials and empty the 'waste' vials from the autosampler tower. Rinse each vial twice with clean methylene chloride, then refill the 'solvent' vial with clean methylene chloride. Rinse the vial cap inserts thoroughly with dichloromethane before capping the vials and returning them to the autosampler. The needle-guide should also be removed from the foot of the autosampler tower and rinsed thoroughly with methylene chloride. The septum and liner should be changed every 2-3 days, before analysis of the opening CCV.

DAILY SEQUENCE

Each sequence should begin with an Instrument Blank and a Carbon Marker (Retention Time verification) Mix followed by a Continuing Calibration Verification (CCV) standard for diesel. Once the opening CCV's have passed acceptance criteria (%D $\leq$ 15%), sample extracts may be added to the instrument sequence. Additional diesel CCV's must be analyzed after every ten samples, not including known QC, but including any unknown non-extracted test samples, and at the end of the sequence. The concentration of the CCV 's must be varied within the calibration range, excluding the highest or lowest points. Additional fuel CCV's (motor oil, jet fuel, or any other fuels that you need to include for the samples in the sequence) must bracket any samples requiring those fuels and the method blank associated with those samples. Additional Alkane (RT) Mix standards must be analyzed every 12 hours at minimum, or with every 2nd set of bracketing CCV's.

Writing an EZChrom Sequence (see Appendix_10 for EZChrom instructions)

Various user reports are automatically produced after the run is complete. This automation is based on the type of sample being analyzed, the sample number, the LIMS identification of any associated calibration or spiking standards, the batch number, and any applicable dilution factors. This data must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information and should be written into the sequence as follows:

Primer
IB
CMARKER,S#
CCV,S#,DSL_<##>
CCV,S#,MO_<##>
MB,QC#, <b#>
LCS,QC#,<b#>
Sample#,<b#>,IDF
MS,QC#,<b#>
MSD,QC#,<b#>

Where: S# is the LIMS S# of the standard used (ie: 06S1365) s12545,
is the concentration of the standard (mg/L)
QC# is the LIMS ID of the batch QC sample (ie: QC169359)
b# is the LIMS batch number (ie: 124536)

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IDF is the instrument dilution factor for the sample (ie: "1000x" or similar), used only if necessary, otherwise left null
PDF is the prep dilution factor for the sample (ie: 0.02 or similar)
Samplenum is the LIMS sample number (ie: 160961-005)

Silica Gel: If a silica-gel cleanup has been performed on the sample or QC, add an "sg" to the name of the sample or batch QC number (ie: 170269-003sg, or QC138963sg).

Examples of EZChrom file names include:

Sample files:	Julian-date/a(or b)/<file number> (ie: 004b012)
Process files:	asur115.met

For data file names, the first three characters of the data file name are the Julian day; the fourth character is A for Channel A (if the front injector port is used) or B for Channel B (for the back injection port) data files. The files are written to the G:\GCxx\CHA or CHB subdirectory, where xx is the GC number.

Example: The complete path for the 12th injection onto GC15b on January 4 would be G:\GC15\CHB\004b012.

For process files, the first letter is the channel number, followed by an identification of what the method does ('sur' for surrogate, 'teh' for fuels), and the Julian date of the sequence containing the relevant files. If the process (method) file is updated more than once on the same Julian sequence, a letter will be appended to the file name (ie: asur115a.met).

Instrument Sequence
The following is a typical sequence, only the diesel CCV's are required for analysis unless others are requested:

1. PRIMER (any standard or dichloromethane)
2. CCB IB - Instrument blank (clean dichloromethane)
3. CMARKER (Alkane Mix for RT verification)
4. 250 mg/L JP 5 standard - (if requested)
5. 500 mg/L Diesel standard
6. 500 mg/L Motor Oil standard - (if requested)
7. Method Blank
8. LCS or Blank Spike
9. Matrix Spike or Blank Spike Duplicate
10. Matrix Spike Duplicate
11-20. Unknown Samples
21. JP 5 CCV
22. Diesel CCV
23. Motor Oil CCV
24-34. 10 samples
35. CMARKER (Alkane Mix for RT verification)

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36. JP 5 CCV
37. Diesel CCV
38. Motor Oil CCV

The sequence must end with a CCV and an Alkane Mix regardless of the number of samples analyzed. If a new sequence is beginning immediately after finishing one, an alkane mix does not need to run at the end of the closing sequence, as long as one ran with the previous bracket's ccvs; the opening sequence's alkane mix will run within 12 hours of the previous alkane mix.

Although the current SW-846 methods allow up to twenty runs between CCV's, C&T typically runs CCV's after every ten samples to meet the additional SW-846 requirement that no more than 12 hours should elapse between CCV's and to reduce the number of reanalyses caused by failing CCV's.

Note: If the instrument will be running unattended or overnight, it is a good idea to load two of each CCV standards for each bracket, to reduce the number of samples that have to be reanalyzed due to an injection error.

1.) Primer

If the instrument has been idle for several hours, it's a good idea to start the instrument with an injection of dichloromethane or any standard, to rinse the injection system and anything (column bleed, contamination, etc.) that may have gathered in the injection port, column, or detector.

2.) CMARKER - Alkane Mix (Retention Time Verification standard)

Carbon Marker (retention-time verification) standards must be analyzed every twelve hours and at the end of the sequence; generally loading these standards with every other CCV bracket will meet this requirement. The carbon-markers that define the ranges needed for the associated samples must fall within the expected retention time for those carbon ranges; if the RT's drift outside the RT windows, the method must be updated and all associated data reprocessed against the corrected retention times. The method must be updated and the data will only be re-analyzed when it's impacted by the retention shift.

If the Carbon Markers routinely fall outside their expected retention window, do a 72-hour RT study to determine if the windows should be changed; see Appendix_4 for further discussion of RT windows.

3.) Hydrocarbon Continuing Calibration Verification (CCV):

After the instrument blank has shown that the instrument is not contaminated, analyze a diesel CCV (Continuing Calibration Verification) standard to verify that the response of the instrument has not changed significantly and that the curve may still be used to quantitate sample results. Use CCV standards at the mid-levels of the calibration curve for all other fuels but diesel; do not use either the highest or lowest point. For diesel CCVs, the CCV levels are alternated.

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3.1) Review the job sheets to decide what CCV standards to analyze, keeping in mind that the concentrations must be alternated across the mid-levels of the calibration curve.

Note: The USACE recommends that the CCV's be the same standards, or from the same source, as the ICAL standards in order to more readily determine which problems are due to changing instrument conditions and are not due to differences between standards.

3.2) Load CCV standards after every ten samples and at the end of the sequence, not including batch QC and excluding instrument blanks and other standards in the count.

LIMs identifies samples that are associated with the DoD QSM 4.1 requirements, other DoD requirements or commercial clients. It identifies all project specific criteria that are reviewed by the analyst before sample analysis.

For sequences containing samples that are not associated with requirements from DoD QSM 4.1, If the instrument is running unattended or overnight, it is a good idea to load two CCV standards for each bracket, to reduce the number of samples that have to be reanalyzed due to an injection error. Type in the sequence with an "x" stype for the second CCV so that only the first CCV will automatically process.

For sequences where both DoD QSM 4.1 samples and non-DoD QSM 4.1 samples are analyzed, both sets of CCVs will be run and integrated and both CCVs must pass the DoD QSM 4.1 criteria. If the entire sequence contains DoD QSM 4.1 samples only, then only one CCV will be analyzed.

3.3) Analyze the standards using the same data acquisition method as for the samples, typing "CCV," before the working standard number, so that LIMS will automatically generate a CCV summary (Continuing Calibration Verification summary), which compares the calculated concentrations from this run to the known concentrations of the standard.

3.4) Integrate the fuel chromatogram. Manual integrations must be consistently applied to standards and samples; see Appendix_7 for integration events & examples.

3.5) Scale the chromatogram so that the baseline corrections and integration flags are clearly visible. Scale the chromatogram so the entirety of the client's extract is visible. If the extract has low level hydrocarbons present, make sure not to zoom in too closely (around 400 mV), and try to be consistent with scaling throughout a client's job. The integration flags and baseline corrections are present on the raw method data and not on the chromatogram.

3.6) Print the data to LIMS//Ezchrom_capture then examine the CCV summary to determine if the %D is $\leq$ 15% and the CCV is acceptable.

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Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), and the method 8015D requirement for the ICV & CCV %D is ≤ 20%.

3.7) If the acceptance criteria are not met, examine the integration to verify that each peak was correctly integrated. See Appendix 7 for examples of acceptable integration.

Unsubstantiated alteration of peak integration solely to pass calibration or QC criteria is illegal and is grounds for immediate termination.

3.8) If the acceptance criteria are not met, analyze another CCV standard. If the second analysis of the standard fails to meet the criteria, perform instrument maintenance and analyze another CCV; if the CCV is still not passing then recalibrate.

If two CCV's were analyzed, examine the first one against the acceptance criteria; if it fails, "x" out the first CCV, change the second to stype "CCV" and process the data from the second CCV.

3.9) If any of the fuel CCV's fail acceptance criteria, data may be reportable based on the following criteria:

a.) If the failing analyte is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b.) If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c.) If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.

d.) If the compound fails the %D criterion due to a low response, the sample must be reanalyzed, as a low bias may result in false negatives.

Load additional CCV's after every ten samples and at the end of the sequence. The standard concentration used for the CCV should be alternated for diesel over the course of the sequence. See Appendix 1 for calculation of %D.

Recommendation: Although SW-846 methods 8000 and 8015 do not require, or even discuss, the analysis of continuing calibration blanks (CCBs/IBs), at least one instrument blank should be analyzed, usually at the beginning of each sequence, with the first set of CCVs, to demonstrate that the analytical system is not contributing to the analytical results. If any target compounds are detected in this blank at levels greater than the reporting limit, determine the source of the contamination and correct the problem before continuing with sample analysis.

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4.) Surrogate Continuing Calibration Verification (CCV):
Compare the surrogate concentrations in the first CCV standard to the expected concentration. If the result differs from the expected concentration by more than 15%, check the instrument for possible problems. Low surrogate recovery with obvious peak tailing is a sign of inlet activity/contamination or column degradation; changing septum, liner and/or cutting a loop off of the guard column usually corrects this situation. If no leaks are discovered and instrument maintenance does not produce an acceptable surrogate CCV, recalibrate the surrogate.

Note: **Acceptable sample data must be bracketed by acceptable surrogate CCV's.**

Note: The method 8015D requirement for the ICV & CCV %D is ≤ 20%, however CA-DHS has not yet recognized 8015D, so C&T must continue to meet the 8015B requirement.

5.) Prepare the Sample and Batch QC Extracts for Analysis

5.1) Remove the extracts from the extraction lab refrigerator and let the extracts warm to room temperature then aliquot approximately 1mL of extract into an autosampler vial.

5.2) If dilutions are required, see Appendix_3 for instructions on preparing the dilutions.

5.3) Place the samples and standards on the autosampler tray as described above, beginning with the lightest colored extracts followed by more highly colored or viscous extracts. If a highly colored or viscous extract needs to run before a lighter one, then run a CCB/IB between the two. Also, try not to run a CCB/IB directly before a CCV.

5.4) Enter the sequence information (sample numbers, batch numbers, any applicable instrument dilution factors) into the EZChrom sequence. See Appendix_10 for instruction on using EZChrom to set up a sequence.

5.5) Sequences are verified in LIMs and ask another analyst to review the entry against the vials in the autosampler tray. Any errors in the sequence entry must be corrected before processing and reporting the sequence data.

QUALITATIVE ANALYSIS

1.) Integration
Manual Integrations are required whenever chromatograms contain unresolved components that raise the baseline. Since these components must be included in the area sum, reprocess the raw files using forced baselines (see Appendix_7 for EZchrom "integration events") and save the modified result file. The baseline event should be the same as that used in the calibration for that hydrocarbon. Often, components other than the fuels of interest make the use of certain baseline projections impractical. In this case, the analyst's judgment must weigh heavily in correctly integrating the sample chromatogram. *Baselines are drawn from where the baseline starts to deviate from the trend to a second inflection*

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point in the chromatogram, or to the end of the chromatogram if there is no second inflection point.

WARNING: Unsubstantiated alteration of peak integration solely to pass QC criteria (ie: calibration, surrogate) is illegal and is grounds for immediate termination of employment.

2.) Fuel Pattern Recognition

Fuels are not discrete chemicals but are comprised of many related compounds and the composition of the fuel mixture is dependent upon multiple variables. The standards used for quantitation are only *examples* of each fuel.

In general, lighter hydrocarbons will elute before heavier hydrocarbons however various fuel patterns overlap. For example, Jet Fuels will typically be represented by hydrocarbons between C10-C16 while Diesel will elute somewhere between C10-C36. Note that higher fuel concentrations will spill over into adjoining ranges. For example, a high motor oil content will also contain a smaller fraction of hydrocarbons in the diesel range; these should be reported as diesel because, even though the chromatogram may exactly match a motor oil standard, those hydrocarbons within the jet fuel or diesel ranges will still behave in the environment in the same manner as those originating in a diesel mixture.

For our clients to adequately treat, or remediate, environmental contamination, they may need additional information aside from simply the concentration of fuel in each range. We therefore provide the sample chromatograms and indicate through “flagging” whether or not the chromatogram is representative of the fuel being quantitated; see Appendix 6 for example fuel chromatograms). Some of the things to consider when examining fuel chromatograms include:

- The lack of sharp, well-defined alkane peaks does not necessarily indicate that the chromatogram does not represent the fuel. After the fuel has been processed by the refinery, the aliphatic (straight-chain) compounds begin reacting with the many other hydrocarbons present, reducing the number of individual peaks into an undifferentiated ‘blob’ or ‘hump-o-gram’.

- Hydrocarbons migrate through soil at varying rates. The lightest hydrocarbons tend to disperse more quickly, changing the profile of the remaining fuel. This is often referred to as “weathering”.

- The composition of the hydrocarbon mixture depends on the temperature programming used by the refinery during the distillation process and so varies by supplier.

- The composition is also changed seasonally, and may be specific to a certain geographic location (Alaska’s diesel looks like California’s kerosene or jet fuel).

To be considered representative of the fuel, the chromatogram should have the same *basic* shape as the standard, but doesn’t have to exactly match the standard.

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3.) Qualifier Flags

Compare the sample chromatogram with the CCV's for the requested fuel hydrocarbon patterns. *What we're looking for here is consistency*. If the chromatogram resembles the standard (to resemble the chromatogram should have the same *basic* shape as the standard, but doesn't have to exactly match the standard), don't apply any qualifier flag. The lack of sharp alkane peaks is not necessarily an indication that the hydrocarbon does not resemble the standard (fuel may be 'weathered'). If the sample chromatogram does not resemble the standard, flag the data with a "Y". See Appenix_6 for example chromatograms.

If the sample chromatogram contains only a single peak (or several peaks, but no hydrocarbon 'hump'), flag the sample data with only a "YZ"-flag. If the sample chromatogram contains a fuel pattern but the quantitation is being a *significantly biased* by a single peak (or peaks), flag the sample data "Z".

In summary:

Y Does not resemble the requested standard.

Z Resembles the requested standard, but also includes a single peak that significantly biases the quantitation.

YZ Quantitation based only on a single peak or peaks.

QUANTITATIVE ANALYSIS

Analyte quantitation is done using the external standard technique. Quantitation is determined by the total area of the fuel within the selected retention time range to the initial calibration curve for that compound, with adjustments for the sample preparation concentration factor and instrument dilution factor. The start and end time of the area is determined by the beginning or ending retention time of the windows identified in the carbon marker (i.e. use the retention time of the apex of the peak then +/- .9 which is ½ the window). If the surrogate peak elutes within the carbon-range of the fuel, the area of the surrogate peak is subtracted from the total area before the concentration is calculated. See Appendix_1 for example calculations and Appendix 9 for the <u>initial calibration</u> procedure. Concentrations are expressed as micrograms per liter (µg/L) or milligrams kilogram (mg/kg).

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

Evaluate the Batch QC

For every batch of 20 samples (or less) analyzed, a Method Blank (MB), a Laboratory Control Sample (LCS), a matrix spike (MS) and duplicate (MSD) are extracted and analyzed. If insufficient sample volume was submitted for matrix QC, a blank spike (BS) and blank spike duplicate (BSD) are extracted in place of the LCS/ MS/ MSD.

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Note: Project-specific quality assurance project plans (QAPP's) may contain different requirements than those listed in this SOP. If so, the QAPP requirements supersede this SOP for all samples related to that project.

1.) Method Blank

A method blank is extracted with each batch of samples to verify that contamination was not introduced by the extraction glassware or reagents. No fuels should be detected in the method blank above the reporting limit, however if contamination is detected, the blank should be reanalyzed; if contamination is again detected, use the following steps to determine the corrective action required:

a.) If the concentration of the contaminant is below the reporting limit but above 1/2 of the reporting limit, document the contamination and potential for a slight high bias on the batch sequence summary and the data review checklist. Report the data without reanalysis as long as the contaminant does not resemble any low levels hits in the batch's samples; if so, reanalyze and possibly re-extract if it is believed that the hit was caused by a consistent contamination in the batch.

b.) If the fuel found in the method blank was not detected in the associated samples, the data may be reported and the problem narrated. Initiate a Corrective Action Report (CAR) immediately so that re-extraction can begin within the extraction holding time, if necessary.

c.) If the fuel found the method blank was also detected in the associated samples, but the level in the samples is greater than 10x the level in the method blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis. Initiate a Corrective Action Report (CAR) immediately so that re-extraction can begin within the extraction holding time, if necessary.

d.) If the fuel detected in the method blank was also detected in the associated samples, but at levels less than 10x the level in the method blank, and reanalysis confirms the problem, the samples containing the contaminant must be re-extracted and reanalyzed. Initiate a Corrective Action Report (CAR) immediately so that re-extraction can begin within the extraction holding time, if necessary.

2.) Laboratory Control Sample (LCS) or Blank Spike/ Blank Spike Duplicate (BS/BSD):

Laboratory Control Samples are clean aliquots of DI water or sodium sulfate that are fortified or "spiked" with a known amount of diesel; the spike concentration should be in the lower half of the calibration range. The LCS or BS/BSD is then extracted and analyzed with each batch of samples to demonstrate the performance of the extraction and analysis in the absence of matrix interferences. In-house Acceptance Criteria are specified in the associated SOP 'TEH Laboratory Control Limits, Table-1'. These limits are generated semi-annually, using control charts.

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Note: Laboratory Control Samples are physically identical to Blank Spikes, however if a spike is identified in LIMS as a "BS", LIMS will look for a BSD.

2.1) In the LIMS Review-App, select "Review", then "Batch #" to bring up the "reduced" LCS. This lists the results for each carbon range and compares the results to the tightest set of QC limits that are applicable to the jobs in that batch.

2.2) Review the recoveries to determine if the spike is acceptable for all jobs in the batch.

2.3) If either recovery or the RPD (for a BS/BSD) is outside acceptance limits, reanalyze those extracts.

2.4) If the spikes are within acceptance limits upon reanalysis, report the data without further action.

2.5) If reanalysis confirms the failure, initiate a corrective action report (CAR). Review the data on a job-specific basis to identify which jobs are affected by the failure and use the following steps to determine the appropriate corrective action:

a.) If a high recovery is observed but diesel was not detected in the associated samples, report the data without re-extraction, as the potential high bias does not affect the sample results.

b.) If a high recovery is observed and samples contain diesel at levels above the reporting limit, those samples must be re-extracted.

c.) If a high RPD is observed but the recoveries are within acceptance limits and diesel was not detected in any of the samples, note the failure on the Data Review Checklist and report the data without re-extraction, as the lack of good precision data does not affect ND samples.

d.) If a high RPD is observed and diesel was detected in the samples at levels above the reporting limits, those samples must be re-extracted.

e.) If low surrogate recovery is observed, the associated samples must be re-extracted.

f.) If low diesel recovery is observed, re-extract all samples in the batch.

2.6) If a sample must be re-extracted and the holding time has expired, the client's Project Manager should log the sample in as an alias and have the samples re-extracted as the new sample number, so that both results may be reported. If the sample is still within holding time, re-extract and reanalyze the sample under the original sample number.

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3.) Matrix Spike/ Matrix Spike Duplicate (MS/MSD):

Matrix spikes are real-world samples that have been spiked with a known amount of diesel then extracted with that batch of samples to demonstrate the accuracy (recovery) and precision (RPD) of the analysis in real-world samples. In-house Acceptance Criteria are specified in the associated SOP 'TEH Laboratory Control Limits, Table-1'. These limits are generated semi-annually, using control charts.

Review the MS/MSD data. If either the recoveries or RPD fail criteria, use the following steps to determine whether or not the data can be reported:

a.) If the diesel concentration in the native (non-spiked aliquot) sample is greater than the linear range and the sample needs to be rerun for just that compound, report the MS/MSD with a LIMS-flag of ">LR" on those recoveries without reanalysis.

b.) If the diesel concentration in the native sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a ">LR" flag to those recoveries. Report the data without reanalysis.

c.) If the diesel concentration in the native sample is greater than 4x the spiking level, LIMS will apply a "NM" (for "Not Meaningful") flag to those recoveries. Report the data without reanalysis.

Note: If the concentration of a target compound is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

d.) If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

e.) If the recoveries fail due to obvious chromatographic interference (ie: coelution of other analytes with the spike compounds), narrate the failure on the Data Review Checklist and report the data without reanalysis.

f.) If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

Note: If the sample used for matrix QC must be analyzed at a 10x or greater dilution, don't analyze the MS/MSD, as the sample concentration will be >4x the spiking level and the spike results will therefore be considered "not meaningful". An MS/MSD will be analyzed/re-analyzed if the client has requested the associated MSS.

After the batch QC samples have been reviewed and deemed acceptable, assemble the Batch QC folder and complete a Batch Review Checklist. Submit this package to the Department Manager or a QC Chemist for review and approval.

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Evaluate the Sample Results

After the standards and batch QC results have been examined to verify that the sequence and batch can be reported, and the sample chromatograms integrated, examine the data for each sample to determine if the results are complete or if reruns or re-extractions are required.

4.) Surrogates

Surrogate compounds are chemically similar to the target analytes but are compounds not found in actual samples. These compounds are added, prior to extraction, to every sample, method blank, and spike to monitor the efficiency of the extraction for that sample. In-house Surrogate Acceptance Criteria are specified in the associated SOP 'TEH Laboratory Control Limits, Table-1'. These limits are generated semi-annually, using control charts.

After each sample is integrated and sent to LIMS, LIMS will automatically generate a user report with the surrogate criteria for that sample and flag any failing recoveries. Evaluate the surrogate recoveries for all samples, method blanks, and spikes. If the extract was diluted by a factor of 10 or more, the surrogate is considered diluted out and LIMS will place a "DO" flag on the user report and final forms.

If a surrogate recovery is outside QC limits, verify that the prep information (LIMS S#, amount, and concentration of surrogate added, sample weight/ volume, extract volume, and instrument dilution factors) is correct. If any of these are incorrect, fix the entry and reprocess the data. If the prep entry was correct, the sample must be reanalyzed, on another instrument if possible. If the surrogates again fail, determine whether reanalysis is required using the following criteria:

a.) If a high recovery is observed but no target analytes were detected above the reporting limit in the sample, note the failure on the 'Data Review Checklist' and report the data without reanalysis, since the possible high bias will not affect sample results.

b.) If a high recovery is observed and matrix interference is the likely cause (ie: the sample chromatogram also contains significant alkane peaks in the region of the surrogate, or the surrogate peak shows a distinct shoulder), discuss the failure with the Group Leader or QC Chemist, and then report the data with narration of the failure, namely "co-elution".

c.) If a high recovery is observed and target analytes were detected, and there is no obvious chromatographic interference, the sample must be reanalyzed. If the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. If the same surrogate fails criteria after re-extraction it is deemed to be matrix effect. Include both sets of data in the package and note the situation in the case narrative.

d.) If a low recovery is observed for any surrogate and there is no obvious chromatographic interference, or documented historical site matrix interference, the sample must be reanalyzed. If the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. If the

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same surrogate fails criteria after re-extraction it is deemed to be matrix effect. Include both sets of data in the package and note the situation in the case narrative.

If a sample must be re-extracted and the holding time has expired, the client's Project Manager must log the sample into LIMS as an alias and have the sample re-extracted as the new sample number. If the sample is still within holding time, the sample should be re-extracted under the original sample number.

If upon re-extraction, the surrogate recovery is again outside limits, note the matrix effect as "confirmed matrix interference" on the User Report and the Data Review Checklist, otherwise report the data with passing surrogate recovery.

Note: Project-specific quality assurance project plans (QAPPs) may require batch control based on different control limits, in which case the project requirements supersede this SOP for all samples related to that project.

5.) Sample Results

Once the chromatogram is integrated and the data sent to LIMS, LIMS automatically generates a user report. Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

If the concentration in any sample exceeds that of the most concentrated standard, LIMS will apply a ">LR" (greater than linear range) flag to the result. Dilute the extract and reanalyze. Diluted sample concentrations must be above the low-CCV level and preferably in the upper half of the calibration range. Dilute to the middle half of the linear range, and to the lower half if the sample needs to run on the 6890 or 7890 instruments and is the sample has "heavy" elution.

If high levels of hydrocarbons are known or suspected, analyze an instrument blank immediately following the sample to prevent carryover into the next sample. If very high levels are detected in a sample and an instrument blank was not analyzed immediately after the high-level sample, examine the data of the subsequent samples to determine whether carry-over may have contributed to the sample results. If carry-over is suspected, reanalyze the sample to confirm the absence of carry-over contributions.

If a silica gel cleanup was performed on any of the sample extracts, the method blank and LCS/BS/BSD must also be silica-gelled; if the native sample used for the MS/MSD was silica-gelled, the cleanup must also be performed on the MS/MSD extracts. If a client has requested results be reported for both the original and silica-gelled fractions ("pre- and post"), the batch QC for both fractions must be reported. If a client has requested silica-gel cleanup only on those samples containing reportable levels of extractable hydrocarbons ("sg if hit"), examine the sample data to identify which samples should be cleaned up. Write the request for silica gel cleanup on the "silica gel request white-board".

6.) Dilutions

If the concentration in any extract exceeds that of the most concentrated standard, the extract must be diluted and reanalyzed to bring the response into the calibration range.

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Diluted sample concentrations must be above the low-CCV level and preferably in the upper half of the calibration range. See Appendix 3 for preparing various dilutions.

If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

DATA REVIEW & DOCUMENTATION

Review the job sheet to make sure that any special client needs are addressed (for example, CCI needs to be on one instrument, and ITSI Hunter's Point requires special flagging), that the job was logged in properly and has the correct compound list.

Check to make sure the correct clean-up version of samples and QC has run and are ready to report. Finally, search for any CARS associated with job.

Once you know what you need to report, begin by using the C&T search tool and type in the Job#, Product ("gcsv"), and Matrix. This will bring up sample data in LIMS.

In the top right, use the "Review" pull down menu and select the job you would like to report.

Log in if necessary.

Important! You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

Review the LIMS generated user reports, the data reduction quantitation reports, and the chromatograms to ensure that the correct carbon ranges, qualifier flags, dilution factors and results are reported. The user report must be initialed and dated by the analyst reviewing and approving the data for that sample.

Complete and sign the "Data Package Review and Narrative" checklist on line. The completed data package consists of this checklist, C&T Job sheet, and LIMS "Form 1's" (sample and batch QC results). The QC package includes the ICAL summary, CCV summaries, Method Blank summary, LCS/ MS/ MSD summaries, Extraction Batch sheets (including silica gel cleanup logs, if applicable), and sequence files, and any associated Corrective Action Reports.

Submit the data package to the Group Leader, Department Manager, or QC Chemist for second-party review by signing the top of the job in LIMS' review application. Any changes made by the second-party reviewer must be individually initialed and dated by the reviewer. The second party reviewer must initial and date each user report, make any additional comments on the case narrative, and initial and date the completed checklist.

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POLLUTION PREVENTION
Direct the split vent and septum purge lines through a carbon trap in order to reduce solvent emissions into the laboratory. Prepare only sufficient standard volume to use within the shelf-life of the standard to reduce the volume of waste generated by the laboratory.

WASTE DISPOSAL
All sample extracts should be stored in the Delfield refrigerator in the extraction lab. The extracts should be retained for a minimum of 40 days after extraction. After 40 days they should be included in the '$MeCl_2$' waste stream.

Standards stored in scintillation vials are included in the '$MeCl_2$' waste stream, after expiration of the standard expiration date. Autosampler vials containing standards or extracts should be taken to Sample Control for eventual disposal in the 'Solvent/ Solid' waste stream.

REVISION HISTORY
This is rev 17 rev 16 was changed as follows:

- On Page 13 added: The start and end time of the area is determined by the beginning or ending retention time of the windows identified in the carbon marker (i.e. use the retention time of the apex of the peak then +/- .9 which is ½ the window).
- Added: Volume 1, TNI Standard, EL-V1-2009, September 2009
- Added: DoD Quality Systems Manual, Rev 4.2 October 2010

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APPENDIX_1: CALCULATIONS

CALIBRATION EQUATIONS

Response Factor:

RF = Area of standard / Concentration of standard

Percent Relative Standard Deviation (%RSD):
The %RSD is the quotient of the Standard Deviation of the data set divided by the mean of the data set multiplied by 100.

%RSD = SD/avgRF * 100

Where: AvgRF = Average of the initial calibration RF's for a compound
SD = Standard deviation of RF's for a compound

$$= \text{SQRT}\left(\sum_{i=1}^{n} ((Rf_i - \text{avg } Rf)^2/(n-1)) \right)$$

BATCH QC CALCULATIONS

Percent Recovery (%R):
The recovery is the measured concentration divided by the true concentration of the spike.

%Recovery = (Cf – Cs) / (Cws * Vws/ S) *100

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard
S = Sample weight or volume

Relative Percent Difference (RPD):
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

%RPD = |(Cs - Cdup)| / ((Cs + Cdup)/2) * 100

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

For soil MS/MSD's where the sample weights are not weight-targeted, the expected concentrations will vary with sample weight (because the same volume of spike standard is being added to different weights of sample) and must be accounted for when calculating RPD. This is accomplished by:

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"Scaled" MSD conc = ((MSSc + MSsp) / (MSSc + MSDsp)) * MSDc

Where: MSSc = determined concentration in the unspiked aliquot of sample
MSsp = the amount spiked into the MS
MSDsp = the amount spiked into the MSD
MSDc = determined concentration in the MSD

Then: %RPD = (MS – 'Scaled' MSD) / ((MS + 'scaled' MSD)/2) *100

SAMPLE CALCULATIONS

Concentration of Aqueous Samples (ug/L) = (Ax * A *Vt * Df) / (Rf * Vi * Vs)

Where: Ax = Area response for the analyte in the sample (minus the surrogate area, if applicable)
A = Amount (mass) of calibration standard injected in ng
RF = Average Response factor (area/concentration) from the calibration
Vi = Volume of extract injected in uL
Df = Instrument Dilution Factor, if no Dilution D =1, dimensionless
Vt = Volume of Total Extract in uL.
Vs = Volume of Sample extracted in mL.

Concentration of non-Aqueous Samples (ug/Kg) = (Ax * A * Vt * Df) / (Rf * Vi * W)

Where: Ax = Area response for the analyte in the sample (minus the surrogate area, if applicable)
A = Amount (mass) of calibration standard injected in ng
RF = Average Response factor (Area/Conc) from the calibration
Vi = Volume of extract injected in uL
Df = Instrument Dilution Factor, if no Dilution D =1, dimensionless
Vt = Volume of Total Extract in uL.
W = Mass of Sample extracted in grams.

Moisture Corrected Results
If a client requests results reported on a 'Dry Weight' basis, the concentration is divided by the 'solids', where the solids is (100-%moisture)/100.

Dry Weight Concentration (ug/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

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APPENDIX_2: REAGENTS & STANDARDS

REAGENTS

Dichloromethane: "High Resolution GC' grade. Store at room temperature for up to one year, or until the manufacturer's expiration date, whichever is sooner.

Carrier Gas: Helium, 99.99% or better
Makeup Gas: Nitrogen, 99.99% or better
Detector Gases: Hydrogen, 99.99% or better
Air, compressed (air compressor in attic)

STANDARDS DOCUMENTATION

The standards listed below are those in use at the time this procedure was written. Alternate supplies may be used so long as they are of equivalent quality and all other calibration, quality control, and traceability requirements are met.

Source Standards

Source standards are those purchased from a chemical manufacturer or vendor. For source standards, the LIMS S-name is unique to both the composition (compound list) of the standard *and* to the vendor of that standard. A new S-name must be assigned whenever the composition is changed or when the standard is obtained from a different vendor; the information must then be entered in the "Standard Definitions" table before the new standard is assigned an S#. If you need more details, log into the LIMS browser; follow the 'LAB MENU' link and click on the "New Standards System (March 2005)" link for details on the system.

Certificates of Analysis should be obtained from the vendor of each source standard; each standard should be traceable to NIST. Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is 1 year from date received, or sooner if comparison with check standards indicates a problem. Also, opened, ampulated standards should be discarded within 1 year after opening.

Enter the lot#, date received, and expiration date of each source standard into LIMS immediately upon receipt, using the Standards Menu "Standard Inventory".

Label the Certificate of Analysis with the LIMS S#, date received and expiration date (if not already listed on vial) then file the certificates in the 3-ring binder. Label each vial with the contents, LIMS S#, and expiration date.

Working Standards

Working standards are those prepared at C&T, which should be prepared in Class-A volumetric flasks. For working standards, the LIMS S-name is not unique to the source standard vendor but *is* unique to the compound list and concentrations contained in the working standard; if the concentration or compounds in the working standard changes, a new S-name, compound list and concentrations must be entered in the "Standard

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Definitions" table before the standard is logged in and assigned an S#. It is *very important* to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

Working standards expire 180 days after preparation from the source standards unless any of the source standards expire before 180 days. If any of the source standards expire before 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards. **The expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**.

In the Standards Benchbook, enter the prep date, LIMS S#, concentration and volume of each source standard used, the LIMS S-name, final volume and concentration of the working standard, expiration date, and prep chemist's initials.

In LIMS, enter the prep chemist's initials, prep date, and S# of all source standards used to make the working standard; LIMS will then assign a standard number (S#). If you need more details, log into the LIMS browser; follow the 'LAB MENU' link and click on the "New Standards System (March 2005)" link for details on the system.

Expired standards may be used for trouble-shooting or method development so long as each standard vial is clearly marked "expired" and stored in a well-marked tray containing only expired standards.

COMMON FUEL STANDARDS

Source Standards

Label each vial with the contents, LIMS S#, and expiration date. **These solutions must not be used for calibration after supplier noted expiration dates or sooner.** Expired standards may be stored separately, in a well-marked tray, for use in trouble-shooting or method development. Store neat, liquid standards at 4°C (± 2°C) in Refrigerator # 5 in the extraction lab; store any pre-diluted standards in the freezer at < -10°C. Do not store standards in a refrigerator containing samples or sample extracts.

Source Standards

ANALYTE	Concentration	Supplier & Catalog#	LIMS S-Name
Diesel Composite	50,000 ug/mL	Restek # 31259	DIESEL XHC
Motor Oil 10W-40	100%	Chevron "Supreme"	MO_10W-40
Jet A (commercial jet fuel)	50,000 ug/mL	Restek # 31242	XHCJETA
JP-5 (military jet fuel)	50,000 ug/mL	Restek # 31252	XHCJP-5
JP-8 (military fuel)	50,000 ug/mL	Restek # 31254	JP-8
Kerosene	50,000 ug/mL	Restek # 31256	KER
Bunker C	50,000 ug/mL	Restek # 31248	BUNKERC
Creosote	50,000 ug/mL	Restek # 31838	CREOSOTE_R
Hydraulic Fluid	100%	Envirometrix AccuStandard	64742-54-7
Transmission Fluid	100%	Law Environmental NAT	LAWTRNSOIL

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Working Standards

Prepare working standards by diluting source standards to volume in Dichloromethane in a Class-A volumetric flask. Because the solvent may dissipate over time, these solutions must be discarded after 180 days or sooner if there is a discrepancy when compared with check standards. Also, **the expiration date of a working standard cannot exceed the expiration date of the source standard used to prepare it**. If the 180-day expiration date in LIMS exceeds that of the source standard, the expiration date of the working standard must be advanced to the expiration date of the source standard in LIMS, the benchbook entry, and the vial label. Store working standards in the extraction lab freezer at ≤ -10°C.

DIESEL WORKING STANDARDS

Working Standard & Conc. (µg/mL)	Final Volume in MeCl2	Using Std	Add Std (mL)	LIMS S-Name
Diesel ICAL 7,500	25 mL	DIESEL HXC	3.75	DIESEL7500
Diesel ICAL 5,000	25 mL	DIESEL HXC	2.50	DIESEL5000
Diesel ICAL 1,000	10 mL	DIESEL5000	2.00	DIESEL1000
Diesel ICAL 500	10 mL	DIESEL5000	1.00	DIESEL500
Diesel ICAL 100	10 mL	DIESEL5000	0.20	DIESEL100
Diesel ICAL 10	10 mL	DIESEL5000	0.02	DIESEL10
Diesel ICV 500	200 mL	DIESEL	2.0	DSL500ICV
		TEH10_SURR	4.0	
Diesel CCV 1,000	200 mL	DIESEL	4.0	DSL1000CCV
		TEH10_SURR	4.0	
Diesel CCV 500	200 mL	DIESEL HXC	2.0	DSL500CCV
		TEH10_SURR	4.0	
Diesel CCV 250	200 mL	DIESEL HXC	1.0	DSL250CCV
		TEH10_SURR	4.0	

MOTOR OIL WORKING STANDARDS

Working Standard & Conc. (µg/mL)	Final Volume in MeCl2	Using Std	Add Std (mL)	LIMS S-Name
Motor Oil ICAL 5,000	100 mL	MO_10W-40	0.050	MO_5000STK
Motor Oil ICAL 2,500	50 mL	MO_5000STK	25.0	MO_2500
Motor Oil ICAL 1,000	50 mL	MO_5000STK	10.0	MO_1000
Motor Oil ICAL 500	50 mL	MO_5000STK	5.0	MO_500
Motor Oil ICAL 250	50 mL	MO_5000STK	2.5	MO_250
Motor Oil ICAL 50	50 mL	MO_5000STK	0.50	MO_50
Motor Oil ICV 500	200 mL	MO_10W-40	0.10 g	MO500ICV
		TEH10_SURR	4.0 mL	
Motor Oil CCV 500	200 mL	MO_5000STK	20.0 mL	MO500ICV
		TEH10_SURR	4.0 mL	

JET-A WORKING STANDARDS

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Working Standard & Conc. (µg/mL)	Final Volume in MeCl2	Using Std	Add Std (mL)	LIMS S-Name
Jet-A ICAL 5,000	25 mL	XHCJETA	2.5	JETA5000
Jet-A ICAL 3,000	10 mL	JETA5000	6.0	JETA3000
Jet-A ICAL 1,000	10 mL	JETA5000	2.0	JETA1000
Jet-A ICAL 500	10 mL	JETA5000	1.0	JETA500
Jet-A ICAL 100	10 mL	JETA5000	0.20	JETA100
Jet-A ICAL 10	10 mL	JETA5000	0.020	JETA10
Jet-A CCV 250	100 mL	JETA	0.50	JETACCV250
		TEH10_SURR	2.0	

JP-5 WORKING STANDARDS

Working Standard & Conc. (µg/mL)	Final Volume in MeCl2	Using Std	Add Std (mL)	LIMS S-Name
JP-5 ICAL 2,500	25 mL	XHCJP-5	5.0	JP/5 2500
JP-5 ICAL 1,500	10 mL	JP/5-STOCK	3.0	JP/5 1500
JP-5 ICAL 500	10 mL	JP/5-STOCK	1.0	JP/5 500
JP-5 ICAL 100	10 mL	JP/5-STOCK	0.20	JP/5 100
JP-5 ICAL 10	10 mL	JP/5-STOCK	0.02	JP/5 10
JP-5 Intermediate	100 mL	XHCJP-5	5.0	JP5_STOCK
at 2,500 µg/mL		TEH10_SURR	2.0	
JP-5 CCV 250	200 mL	XHC JP-5	1.0	JP5250CCV
		RESO_TERPHENYL	1.0	

JP-8 WORKING STANDARDS

Working Standard & Conc. (µg/mL)	Final Volume in MeCl2	Using Std	Add Std (mL)	LIMS S-Name
JP-8 Intermediate at 5,000 µg/mL	25 mL	XHCJP-8	2.5	JP85000PPM
JP-8 ICAL 2,500	10 mL	JP85000PPM	5.0	JP82500PPM
JP-8 ICAL 1,500	10 mL	JP85000PPM	3.0	JP81500PPM
JP-8 ICAL 500	10 mL	JP85000PPM	1.0	JP8 500PPM
JP-8 ICAL 100	10 mL	JP85000PPM	0.20	JP8 100PPM
JP-8 ICAL 10	10 mL	JP85000PPM	0.02	JP8 10PPM
JP-8 CCV 250	100 mL	JP-8	0.50	JP8CCV
		RESO_TERPHENYL	0.50	

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Bunker C WORKING STANDARDS

Working Standard & Conc. (µg/mL)	Final Volume in MeCl2	Using Std	Add Std (mL)	LIMS S-Name
Bunker C Stock at 5,000 µg/mL	25 mL	Bunker C	2.5	BUNKC5000
Bunker C ICAL 2,500	10 mL	BUNKC5000	5.0	BUNKC2500
Bunker C ICAL 1,250	10 mL	BUNKC5000	2.5	BUNKC1250
Bunker C ICAL 500	10 mL	BUNKC5000	1.0	BUNKC500
Bunker C ICAL 50	10 mL	BUNKC5000	0.10	BUNKC50
Bunker C CCV 500	100 mL	BUNKC5000	10.0	BUNKC_500
		RESO_TERPHENYL	0.5	

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SURROGATE STANDARD

Label each vial with the contents, LIMS S#, and expiration date. **These standards must not be used for calibration after expiration date or sooner if there is a discrepancy when compared with the check standards.** Expired standards may be stored separately, in a well-marked tray, for use in trouble-shooting or method development.

Source Standard

Hexacosane, 99%
Aldrich catalog # EC211-124-1LIMS S-Name: HEXACO
Store at room temperature in the extraction lab until the manufacturer's expiration date or until the check standards indicate a problem with the source standard.
o-Terphenyl
Restek 2,000.00 ug/mL +/- -0.03%
Store in freezer

Working Standards

Follow the steps below to make the 2,500 ug/mL Intermediate working standard (*LIMS S-Name:* TEH10_SURR):

1.) Weigh 250 mg Hexacosane onto a weighing paper.
2.) Transfer the Hexacosane to a Dichloromethane-rinsed Class A 100mL volumetric flask.
3.) Add about 25 mL 1:1 Dichloromethane:Acetone.
4.) Place in an ultrasonic bath until all of the Hexacosane is dissolved.
5.) Allow the solution to cool and bring to volume with Dichloromethane.
6.) Store this standard in the freezer at < -10°C for up to 180 days.
7.) Use this intermediate standard to make the calibration standards.

To make the calibration standards, remove the intermediate from the refrigerator and allow ample time for the solution to warm to room temperature. Use the Vortex mixer to re-dissolve any Hexacosane that has solidified. Rinse five (5) 50mL Class-A volumetric flasks with dichloromethane, then partially fill them with fresh Dichloromethane. Add the standards as listed below. Cap & invert 3 times to mix, then dilute to volume with Dichloromethane. Store these standards in the freezer at < -10°C for up to 180 days, or until comparison to other standards indicates that the standards should be discarded.

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SURROGATE CALIBRATION STANDARDS

WS Standard & Conc. (µg/mL)	Final Volume (mL) in MeCl2	Using Std	Add Std (mL)	LIMS S-Name
Hexacosane ICAL & OTP 200	50	TEHOTPHEXSURR	1.000	HEX OTP 200 mg/L
Hexacosane ICAL & OTP 100	50	TEHOTPHEXSURR	0.500	HEX OTP 100 mg/L
Hexacosane ICAL & OTP 50	50	TEHOTPHEXSURR	0.250	HEX OTP 50 mg/L
Hexacosane ICAL & OTP 25	50	TEHOTPHEXSURR	0.125	HEX OTP 25 mg/L
Hexacosane ICAL & OTP 10	50	TEHOTPHEXSURR	0.050	HEX OTP 10 mg/L
Hexacosane ICAL & OTP 5	50	TEHOTPHEXSURR	0.025	HEX OTP 5 mg/L

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ALKANE MARKER STANDARDS

Source Standards
Label each vial with the contents, LIMS S#, and expiration date. **These solutions must not be used for calibration after supplier noted expiration dates or sooner if there is a discrepancy when compared with the check standards.** Expired standards may be stored separately, in a well-marked tray, for use in trouble-shooting or method development. Store neat, liquid standards at 4°C (± 2°C) in Refrigerator # 5 in the extraction lab. Store neat, solid standards at room temperature. Do not store standards in a refrigerator containing samples or sample extracts.

Source Standards

ANALYTE	Concentration	Supplier & Catalog#	LIMS S-Name
Alkane Mix (C8-C50)	1 mg/mL	AccuStandard DRH-004S-R1-5x	C8-C50
C-50 (Pentacontane), neat, 250mg/ampule	100%	Supelco 442743	C-50

Working Standards
Prepare working standards by diluting source standards to volume in Dichloromethane in a Class-A volumetric flask. Because the solvent may dissipate over time, these solutions must be discarded after 180 days or sooner if there is a discrepancy when compared with check standards. Also, **the expiration date of a working standard cannot exceed the expiration date of the source standard used to prepare it**. If the 180-day expiration date in LIMS exceeds that of the source standard, the expiration date of the working standard must be advanced to the expiration date of the source standard in LIMS, the benchbook entry, and the vial label. Store working standards in the extraction lab freezer at ≤ -10°C.

ALKANE MIX - WORKING STANDARDS

Working Standard & Conc. (µg/mL)	Final Volume (mL) in MeCl2	Using Source Std	Add Source Std	LIMS S-Name
Alkane Mix (C8-C50) at 25 mg/L	100 mL	CMARKER	2.50 mL	C8-C50
		C-50	2.5 mg	

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OTHER STANDARDS (Bunker C, etc.) _____

Neat solutions of other fuel hydrocarbon mixtures (Bunker Fuel, or client-supplied fuels) are kept at 4°C (± 2°C) in the standards refrigerator # 5 in the extraction lab. To prepare a working standard from a neat standard:

1.) Tare a 100mL Class-A volumetric flask.

2.) Using an analytical (4 decimal) balance, measure the mass of fuel identified in the table below into the volumetric flask.

3.) Add about 25mL dichloromethane and swirl until the neat standard is dissolved into the solvent.

4.) Bring to volume with dichloromethane.

5.) Cap & invert 3 times to mix, allowing sufficient time for mixing during each inversion.

6.) Store all solutions in 40mL VOA vials in the extraction lab freezer at ≤ -10°C. **Discard these standards after six months, or sooner if there is a discrepancy when compared with check standards.**

MISCELLANEOUS STANDARDS

WS Standard & Conc. (µg/mL)	Final Volume in MeCl2	Using S- Std	Add Source Std	LIMS S-Name
Hydraulic Fluid at 1,250 ug/mL	100 mL	EN HYD OIL	0.125 g	HYFL 1250
		TEH10_SURR	2.0 mL	
Transformer Fluid at 1,250 ug/mL	100 mL	LAWTRNSOIL	0.125 g	TRANS_1250
		TEH10_SURR	2.0 mL	

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APPENDIX_3: TEH DILUTIONS

Let the extracts warm to room temperature then prepare the dilution in either an autosampler vial or an insert. See table below for appropriate volumes. Shake the dilution and invert 3 times to mix.

Dilution Factor	Made In	Extract Volume (µL)	MeCl2 Volume (µL)
2x	Insert	100	100
	GC vial	500	500
3x	Insert	50	100
	GC vial	250	500
4x	Insert	50	150
	GC vial	250	750
5x	Insert	40	160
	GC vial	200	800
10x	Insert	20	180
	GC vial	100	900
20x	Insert	10	190
	GC vial	50	950
50x	GC vial	20	980
100x	GC vial	10	990

SERIAL DILUTIONS

If you need to make a >100x dilution, first make the 100x dilution listed above, then make further dilutions, in dichloromethane, using that as an intermediate.

Dilution Factor	Using Primary Dil'n	Made In	Extract Volume (µL)	MeCl2 Volume (µL)
200	100x	Insert	100	100
500	100x	Insert	40	160
1,000	100x	Insert	20	180

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APPENDIX_4: INSTRUMENT CONDITIONS & RETENTION-TIME WINDOWS

Instrument conditions will vary; the conditions listed below should be used as guidelines when setting up a new instrument. The specific conditions applicable to a given instrument are listed in the method software and in instrument maintenance logs.

Carrier Gas:	Helium
Head pressure:	55-60 kPa (about 8 - 9 psig)
Split Vent Flow:	12 - 15 mL/min
Septum Purge Flow:	5 mL/min
Injection Volume:	3 µL
Make up Gas:	Nitrogen at 30 mL/min
Hydrogen pressure:	16 psig
Air pressure:	40 psig
Injector Temperature:	280 °C for HP5890; GC14, GC15, GC17
	310 °C for HP6890; GC26
	325 °C for HP7890; GC27
Detector Temperature:	340 °C

Temperature Program:

Initial Temperature:	50 °C
Initial Time:	1 minute
Ramp:	25 °C/min
Final Temperature:	325°C
Final Time:	8 min.

EPC program (GC15, GC14, GC26, GC27):

Initial pressure:	60 kPa
Initial time:	0.5 min.
Rate:	682.6 kPa/ min.
Final Pressure:	40 kPa
Final time:	0.0 min.
Rate A:	4.0 kPa/ min.
Final Pressure A:	125 kPa
Final time A:	0.5 min.

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"SAFETY SHUT-DOWN" (GC15, GC14, GC26, GC27):
A "safety shut-down" will occur when the EPC (electronic pressure control) can not reach the pressure set-points due to problems with the gas supply. The problem may be due to a cored septum, a faulty valve, someone disconnecting a tank incorrectly, a loose injection port screw, a broken or plugged column, or an empty tank. Investigate and correct the problem before restarting the sequence.

The GC will begin beeping up to 2 minutes before the "safety shut-down" happens. If the problem is immediately corrected, the shut-down may be averted. If this happens, watch the sequence to ensure that it does continue correctly.

To restart after a safety shut-down:

1.) Turn off the GC, HP box, and ICON box.

2.) Reboot by pressing ALT, CTRL, DELETE.
If this doesn't work, try pressing the reset button on the computer.
If this still doesn't work, hard-boot the computer by turning off the power, allowing it to sit for about 15 seconds, then turning the power back on.

3.) Turn on GC, HP box, and ICON box.

4.) On GC key pad enter:
[gold], Inj A Pres, 60, [Enter]
[gold], Inj B Pres, 60, [Enter]
[gold], Aux E Pres, 310, [Enter]
[gold], Aux F Pres, 310, [Enter]

If the key pad entries are not reset, the instrument will again shut down.

Retention Time Windows:
For the TEH analysis, there are two types of retention time windows: the window for the fuel range defined by the carbon markers, and the window for each carbon-marker compound. "Retention Time Windows" are necessary for the carbon-markers because the compounds may not elute at *exactly* the same time during each and every injection, due to slight variations in temperature, flow rate, or injection composition (sample viscosity, compound concentrations), etc. The RT-window is the length of time (width, in minutes, on the chromatogram) during which any peak eluting within the window is presumed to be the analyte of interest. "72-hour RTW Study" is a term often used by auditors to describe statistical analysis of the retention times of standards injected over a 72 hour sequence; theoretically, the RT windows determined by this study can be used for routine analysis, however the studies that C&T has conducted in the past often result in windows that are too narrow for routine use. If this occurs, you may use the default retention time windows of $\pm$ 0.03 minutes as specified in EPA 8000B.

If the RT windows are based on a 72-hour study, the RT windows are defined as plus or minus three times the standard deviation of the absolute retention times for each compound in the

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calibration standard mix as measured over the course of 72 hours. The study must include files collected over 72-hours *without column maintenance*. In the event that a standard deviation is 0.00, or < 0.01 minutes, use the 0.03-minute window (see 8000B).

Absolute Retention Times:
The "absolute" retention time of any compound is the expected time of the compound and is the center of the RT window, which is where the top of the carbon marker should occur. Use the retention time for each Carbon Marker injected during that 12 hour shift to evaluate retention time stability. If any of the standards fall outside their daily or preset fixed retention time windows, the system is out of control. Determine the cause of the problem and correct it, then rerun any samples bracketed by the failing standard.

Method Modification Note: EPA 8000B, Section 7.6.5 suggests updating the absolute retention times each time a new sequence is started. Because the retention times for these compounds are relatively stable, C&T has found it necessary to update the retention times only when performing the initial calibration or when drift is observed.

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APPENDIX_5: MAINTENANCE & TROUBLE-SHOOTING

All instrument maintenance and/or trouble shooting must be documented in the instrument maintenance log. Describe the conditions that led to the maintenance, what maintenance was performed, and whether the maintenance fixed the problem. Date and initial the benchbook entry. If the maintenance was performed by an outside contractor, such as Full Spectrum, place a copy of the work order receipt in the 3-ring maintenance binder located in the Department Managers bookshelf; reference this event in the maintenance benchbook.

PREVENTATIVE MAINTENANCE

1) Septa should be replaced after no more than 100 injections.

2) Liners should be replaced every 2 - 3 days or whenever a CCV is out of limits or you cannot get a clean instrument blank or when surrogate exhibits tailing.

3) Leak check and check the tightness of the capillary column detector adapter nut weekly. Replace the ferrule in the adapter when leaking or you can no longer tighten the fitting and the adapter is still loose.

4) Leak check and check tightness of capillary column ferrule nuts in detector and inlet fittings; they should be snug - do not overtighten.

5) Autosampler syringes should be removed from the autosampler and cleaned weekly. Clean by drawing and expelling a 1:1 vol:vol Dichloromethane/Acetone mixture. After 3 syringe fulls, withdraw the plunger and wipe it with a Kimwipe. Repeat; continue until no more residue is being wiped from the plunger.

6) A C_8 to C_{50} n-Alkane mix should be analyzed whenever a change has been made to the GC system, in order to document baseline performance.

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TROUBLE-SHOOTING

Symptom: **Continuing Calibration Verification out of limits (low)**

Reanalyze the CCV after you have made changes to the GC system (ie: septum/liner change). If the CCV still fails, analyze the C_8 - C_{50} n-Alkane mixture to aid in troubleshooting. If the result from the analysis of the n-Alkane mixture is normal, then recalibrate.

Possible causes:

- Bad CCV solution. Are all fuels out, or only one? If only one is out, it probably is the standard; if both are out, it may be the instrument. If only Hexacosane is out, check for peak tailing. Clipping the column may be required.
- Leak in GC inlet. Replace septum, check column ferrule nut on inlet end for tightness, check inlet insert nut for tightness.
- Dirty inlet liner. Replace liner and O-ring seal.
- Inlet seal leaking or dirty. Replace.
- Leaky syringe. Syringes do wear out. Replace.
- Split vent flow has increased. Check split vent flow for correct flow.
  Caution - incorrect flow may be due to a leak; make sure that the system is leak free before adjusting the split vent flow. Change flows only as a last resort - you will probably have to recalibrate after you change the flow.
- Detector gas flows have changed, especially H_2. Check flows and adjust as required.
  Caution - incorrect flow may be due to a leak; make sure that the system is leak free before adjusting flows. Change flows only as a last resort - you will probably have to recalibrate after you change the flow.

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Symptom: **Continuing Calibration Verification out of limits (high)**

Possible causes:

- CCV solvent has evaporated. Make a new CCV and reanalyze.
- Autosampler not injecting correct amount. HP 7673's will start to fail by intermittently injecting 1 stop (µl) more. Watch the autosampler inject; if it injected the correct amount, was the CCV in limits?
- Split vent flow has decreased. Check split vent flow for correct flow. Caution - incorrect flow may be due to a leak; make sure that the system is leak free before adjusting the split vent flow. Change flows only as a last resort - you will probably have to recalibrate after you change the flow.
- Detector gas flows have changed, especially H_2. Check flows and adjust as required. Caution - incorrect flow may be due to a leak; make sure that the system is leak free before adjusting flows. Change flows only as a last resort - you will probably have to recalibrate after you change the flow.

Symptom: **Dirty instrument blank**

Possible causes:

- Septum Bleed appears as a series of peaks after the hexacosane peak.(See Restek catalog for an example). Septum bleed is most prevalent after the instrument has been sitting idle for a few days. If this is the case, the contamination should dissipate after a few runs. If after a few runs it is still present, replace the septum and liner (often pieces of septum fall into the liner.)
- Semivolatile or nonvolatile sample residues in injector. Remove injector liner and stainless steel seal and swab out injection port with a Q-Tip soaked in Dichloromethane and Hexane. Change septum, injector liner and stainless steel seal. Reanalyze instrument blank.
- Semivolatile or nonvolatile sample residues in column. Remove column from injector and cut off about 1 loop. Reinstall. Reanalyze instrument blank. If instrument blank is still dirty, rinse column and reanalyze instrument blank. See RESTEK literature for column rinsing instructions.

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Symptom: **Early eluting (around C_{10} to C_{20}) alkanes in the carbon-marker mix have low response, or are tailing, or both.**

Possible causes:

- Leak in GC inlet. Replace septum, check column ferrule nut on inlet end for tightness, check inlet insert nut for tightness.
- Improper column insertion distance in inlet liner. Check that the column protrudes between 3 - 7 mm past the front of the ferrule in the capillary column nut.
- Dirty inlet liner. Replace liner and O-ring seal.
- Inlet seal leaking or dirty. Replace.
- Leak in detector fittings or improper column installation in the detector. Check tightness of detector adapter nut. Column should be 1 - 3 mm below top of detector jet.
- Dirty column. Remove column from injector and cut off about 1 loop and reinstall. Reanalyze carbon-markers. If this does not correct the problem, replace the column.

Symptom: **Late eluting (around C_{40} to C_{50}) alkanes in the carbon-marker mix have low response.**

Possible causes:

- Leak in GC inlet. Replace septum, check column ferrule nut on inlet end for tightness, check inlet insert nut for tightness.
- Poor volatilization of higher mole weight compounds. Pyrex wool in inlet liner packed too tightly, or placed too far up or down in liner. Replace liner and reanalyze the carbon-markers.
- Improper column insertion distance in inlet liner. Check that the column protrudes between 3 - 7 mm past the front of the ferrule in the capillary column nut.
- Leak in detector fittings or improper column installation in the detector. Check tightness of detector adapter nut. Column should be 1 - 3 mm below top of detector jet.

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Symptom: **Inconsistent retention times.**

Possible causes:

- Leakage through the septum. You may need to replace the septum more frequently, possibly daily.

Symptom: **Difficulty in igniting FID or flame goes out during elution of solvent.**

Possible causes:

- FID flame tip (jet) dirty or partially plugged - replace. You may have to recalibrate after you replace a jet.

- Improper flow rates of hydrogen and/or air. There should be about a 9 to 1 ratio of air to fuel in order for the flame to quickly ignite. The hydrogen flow can be turned off by using the needle valve at the upper left of the GC control panel. As you hold the ignition button down, slowly open the hydrogen valve. The flame should light with a "pop" sound and remain lit. Fully open the hydrogen valve.

Symptom: **Cannot generate a valid calibration.**

Possible causes:

- Bad calibration solution. Does one calibration level have a much different calibration factor than the rest? This calibration solution may be bad; remake and analyze new solution.

- Dirty column. Remove column from injector and cut off about 1 loop and reinstall. Reanalyze C_8 - C_{50} Alkane (carbon-marker) standard. If this does not correct the problem, replace the column.

Symptom: **Baseline resembles a sine-wave.**

Possible causes:

- Water trap on the air compressor is full and needs to be emptied; the drain may be plugged. (Wave pattern is due to pressure pulse through the detector)

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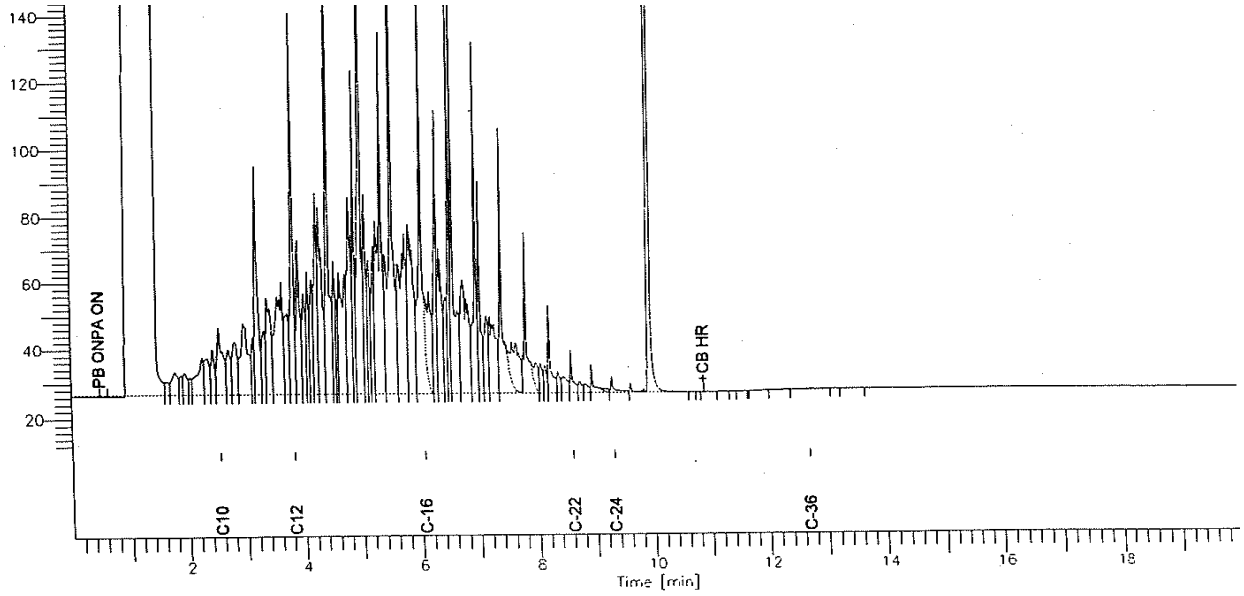
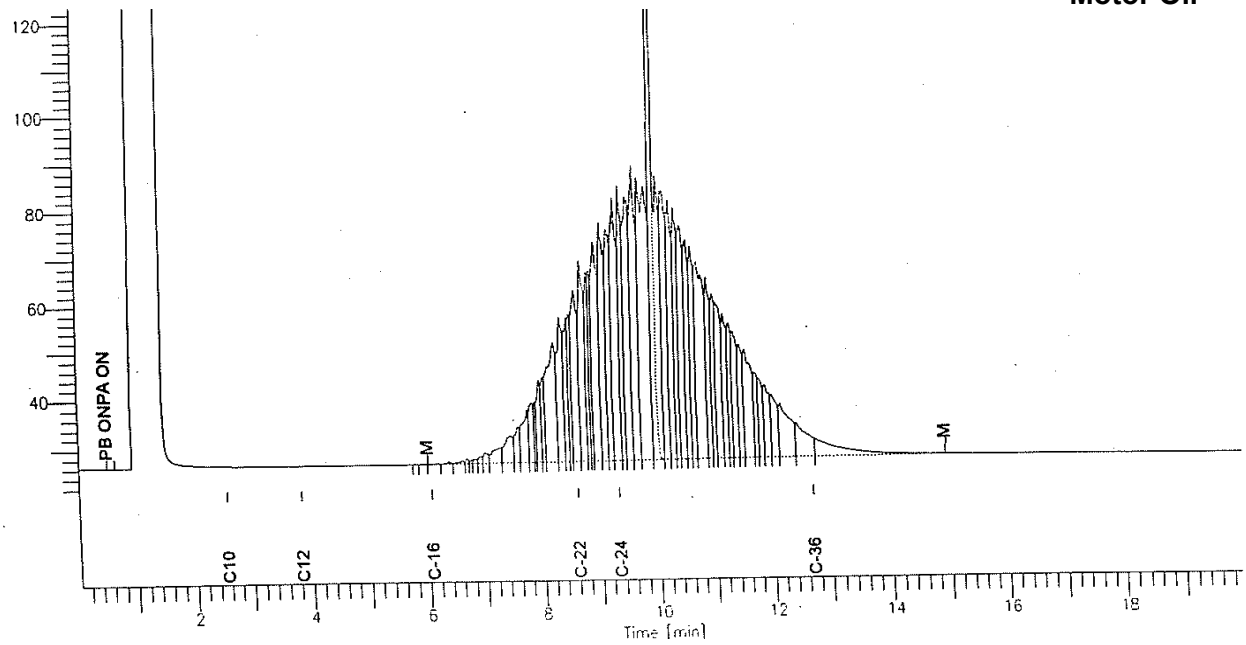
APPENDIX_6: EXAMPLE CHROMATOGRAMS

Diesel



Motor Oil



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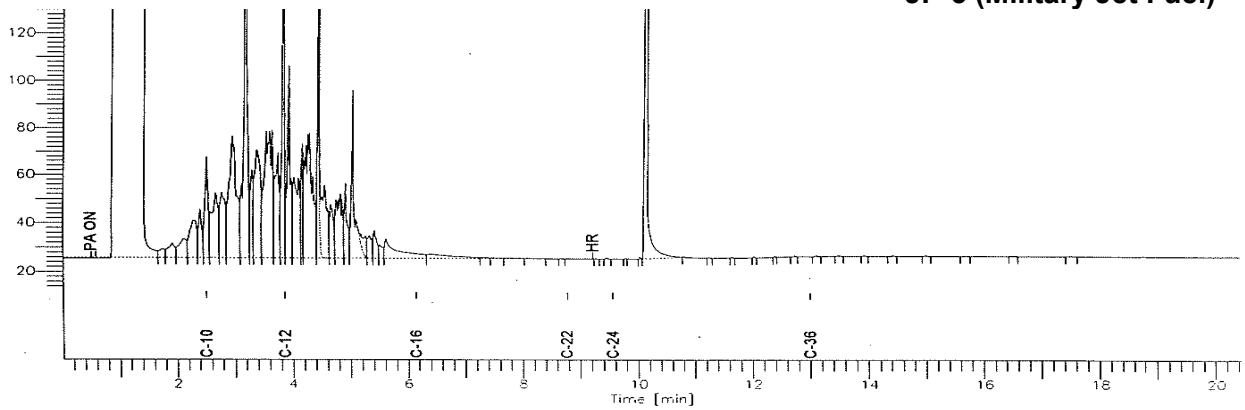
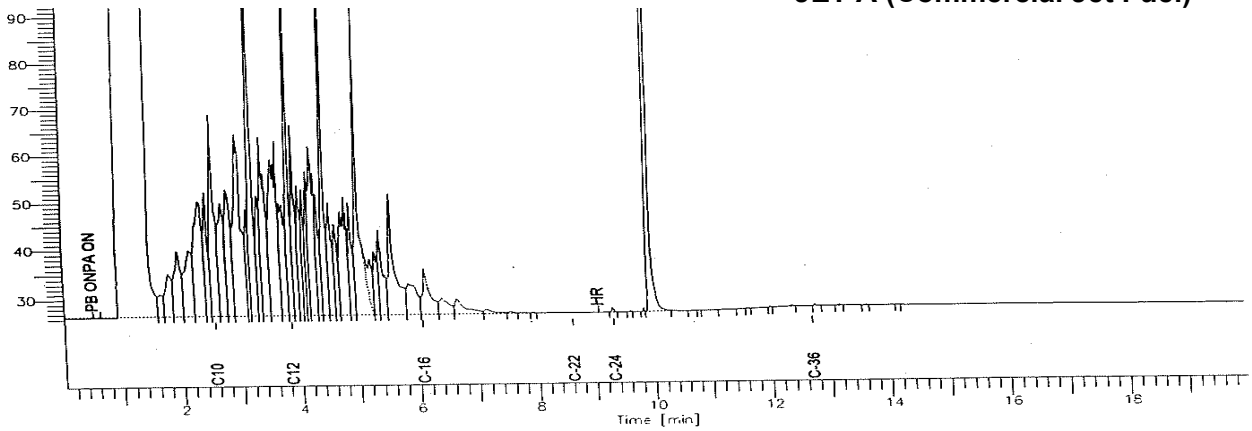
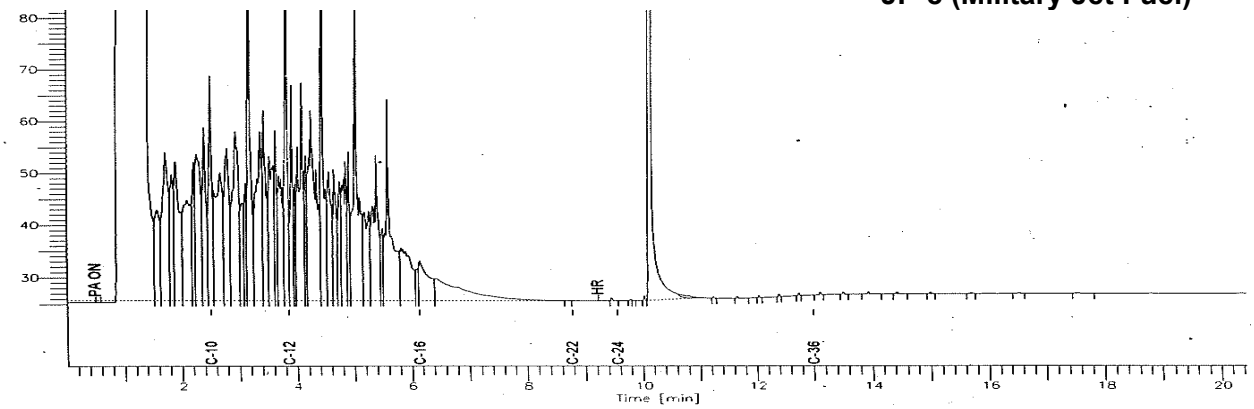
JP-5 (Military Jet Fuel)



JET A (Commercial Jet Fuel)



JP-8 (Military Jet Fuel)



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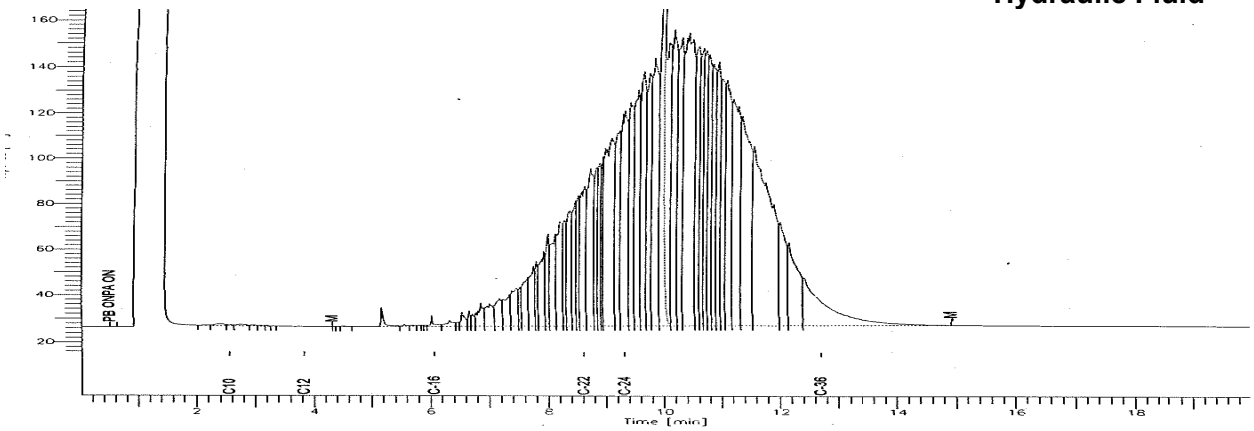
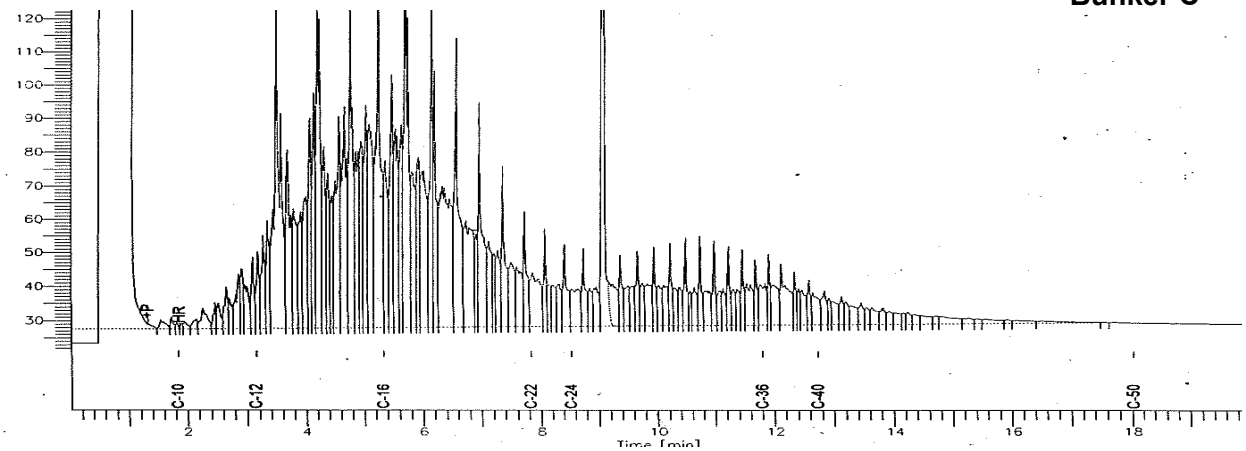
Hydraulic Fluid



Bunker C



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APPENDIX_7: EZCHROM INTEGRATION EVENTS

METHOD DEFINED EVENTS (from EZChrom help files):
Two Integration events are required for each run: Width, and Threshold. These events are used to detect peak start, stop, and apex, and to distinguish true peaks from noise. Default values are entered automatically when you create a new method. If you clear an Integration Event Table, you must enter these events again, otherwise you will get an error message when you analyze the chromatogram.

a.) **Width**
The Width event is used to calculate a value for bunching, or smoothing, the data points before the integration algorithm is applied. Integration works best when there are 20 points across a peak. If a peak is over-sampled (i.e. the sampling frequency was too high), the Width parameter will be used to average the data such that the integration algorithm sees only 20 points across the peak. In setting a Width value graphically, use the narrowest peak in the chromatogram.

A Width event will be applied to a given peak as long as it occurs before or on the apex of the peak.

The Width parameter is only used to correct for over-sampling. It cannot correct for data that was under-sampled (i.e. sampling frequency too low causing fewer than 20 points acquired across the narrowest peak.)

Note: In most circumstances, an initial Width value based on the narrowest peak in the chromatogram will be adequate for proper integration of all peaks. However, a new Width timed event should be entered every time a peak width doubles.

b.) **Threshold**
This parameter is the first derivative, used to allow the integration algorithm to distinguish the start and stop of peaks from baseline noise and drift. When setting the Threshold value graphically, you select a section of baseline. The recommended Threshold value is based on the highest first derivative value determined in that section of the chromatogram.

Note that extreme values of both Width and Threshold (too large or too small) can result in peaks not detected.

c.) **Force Peak Start /Force Peak Stop**
These events are used to force the start or stop of the peak integration to a specific point.

d.) **Reset Baseline -- (no definition from EZChrom)**
This event will cause the baseline to be reset at any selected position. This can result in a discontinuous baseline. The “Reset Baseline at Valley” is more commonly used when manually integrating since it does not give a discontinuous baseline.

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e.) **Valley to Valley**
This event causes the baselines of peaks that are not totally resolved (i.e. do not return to baseline) to be drawn to the minimum point between the peaks. If this event is not used, a baseline is projected to the next point at which the chromatogram returns to baseline, and a perpendicular is dropped for peaks which do not reach baseline.

f.) **Shoulder Sensitivity**
This parameter is used to enable the detection of shoulders on larger peaks. A larger value will decrease shoulder sensitivity, while smaller values increase sensitivity to shoulder peaks. When setting the Shoulder Sensitivity value graphically, you select a section of the baseline. The recommended Shoulder Sensitivity value is based on the highest second derivative value determined in that section of the chromatogram.

g.) **Integration Off**
This event turns off the integration of your chromatogram during the range specified. This event is useful if you are not interested in certain areas of your chromatogram, and do not wish peaks to be reported for that section.

USER-ADDED INTEGRATION EVENTS (from EZChrom help files):

a.) **Manual Peak**
This command allows you to graphically define a peak that was not previously detected. This is convenient when you want to force integration of a peak, but do not want to change your overall integration parameters.

To use this event, click on the Manual Peak button from the Graphical Integration toolbar. Click once on the start of the peak to be defined. Click again on the end of the peak to be defined.

b.) **Reassign Peak**
This event allows you to graphically designate a different peak as the calibrated peak in place of the peak which has been identified. This event does not change the values in the Peak Table.

c.) **Split Peak**
This event is used to force a perpendicular drop-line integration in a peak. The perpendicular will be dropped at the point where the event is inserted.

d.) **Move Baseline Start/ Stop**
These events allow you to move the start or stop of a baseline by clicking and dragging it to a new location.

1. When you select either Move Baseline Start or Move Baseline Stop, you will be prompted to click on the baseline segment you want to modify. The start and end points of the baseline will then appear highlighted with a box.

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2. When you move the cursor to a location within range of the start or stop point, it will turn into an "anchor". Click the left mouse button and "drag" the baseline start-point to the new location, then let go.

3. You can continue to "click and drag" the baseline in this manner until it is in the correct location. Then press the "Esc" key. A dialog box will appear.

4. Click “Analyze now” to add the event to the events table and analyze the chromatogram with the new events.

e.) **Reset Baseline at Valley**

This event will cause the baseline to be reset at the next valley detected after the event. To enter the event graphically, click on the location you want to enter the event, then click the Reset Baseline at Valley button at the bottom of the window.

Note: The event should be placed after the start of the first peak in the cluster; otherwise the start of the peak will be identified as the valley.

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APPENDIX_8: **INITIAL CALIBRATION**
Requirements & Procedure

REQUIREMENTS
An initial calibration curve must be analyzed whenever instrument conditions (temperature programs, flow rates, etc.) have been changed, the detector has been cleaned, or when a new column has been installed. In general, a new calibration curve must be made whenever instrument conditions have been altered, or whenever the continuing calibration verification no longer passes acceptance criteria.

The instrument analytical range must be established by running a minimum of 5 calibration standards, containing the target compound, at levels that bracket the quantitation range (see Appendix_2 for standards information); the lowest standard must be at or below the reporting limit and the highest standard determines the upper end of the quantitation range. The standards must be analyzed in order of increasing concentration.

Points may be dropped if the following criteria are met:

- Using average response, the RSD must be ≤ 20%.
- The highest concentration standard may be omitted so as long as there are at least five points remaining and the remaining highest point defines the top of the calibration range. Any extracts which exceed this response must be diluted and reanalyzed; LIMS will apply an '>LR' flag to any results above the highest ICAL standard.
- The lowest concentration standard may be omitted from curve if, *and only if*, the resulting lowest standard is at or below the reporting limit for samples and there are at least five points remaining.
- Mid-point standards may not be omitted simply to improve the RSD or linear correlation coefficient. They may, however, be reanalyzed if a poor injection is suspected. The reanalysis must occur immediately after the standards, so long as no sample extracts were analyzed since the last standard or blank and all compounds are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria.

The curve must be verified before any sample extracts are run, by analyzing an Initial Calibration Verification (ICV) standard comprised of standards obtained from a different manufacturer than those used to prepare the ICAL standards. If a standard cannot be obtained from a second vendor, or the fuel composition is particular to a specific vendor, an ICV may be prepared from a different lot. *The ICV must meet CCV %D criteria.*

Note: Because fuel composition varies greatly between standard vendors, an ICV is normally analyzed only for the diesel calibration.

See Appendix_1 for calculation of response factors, RSD, and correlation coefficient.

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A carbon-marker standard (C_8 to C_{50}) must be analyzed along with the initial calibration in order to set retention times. *Note:* The State of Arizona requires that the carbon markers be run *after* the fuel standards.

Obtain area sums for each fuel mixture over the retention time range during which at least 85% of the material elutes. Curtis & Tompkins default **carbon ranges** are:

Jet Fuels:	C_{10} to C_{16}
Kerosene:	C_{10} to C_{16}
Diesel:	C_{10} to C_{24}
Motor Oil:	C_{24} to C_{36}

Method Modification: SW-846 Method 8015D lists the carbon-range for diesel as C10-C28, however C&T has historically reported diesel as C10-C24 & continues to do so for data consistency. Also, client project plans may require different carbon ranges; for those clients, the project plan requirements supersede this SOP.

Clients may request the analysis of fuels other than our typical fuels, and may provide a standard of the fuel. It is acceptable in these cases to utilize a single point calibration for the alternate fuel, so long as acceptable diesel CCV's bracket the samples. In these cases a CCV for the client-supplied fuel will be analyzed after every 10 samples and compared to the initial single point calibration. The CCV must be within 15% deviation from the original single-point calibration standard. The concentration of these fuel standards is dependent upon its response, but generally made up at 1250 mg/L. Refer to Appendix 2 for directions on the preparation of standards.

PROCEDURE

1.) Prepare the standards as described in Appendix 2.

2.) Prepare an Initial Calibration Verification (ICV) standard from source standards obtained from a different manufacturer than the ICAL standards.

Note: Because fuel composition varies greatly between standard vendors, an ICV is normally analyzed only for the diesel calibration.

3.) Perform any needed instrument maintenance and run a dichloromethane instrument blank. If any target compound is detected above the reporting limit, run another instrument blank.

4.) Load the alkane "carbon marker" standard. *Note:* The State of Arizona requires that the carbon markers be run *after* the fuel standards.

5.) Load the calibration standards onto the autosampler tray in order of increasing concentration.

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6.) Add instrument blanks before and after the ICAL standards to demonstrate that the low-level standard was not influenced by instrument contamination and that analytes at the high-level concentrations will not carryover into real-world samples.

7.) Load the ICV after the instrument blank that follows the calibration standards. The ICV, prepared from standards obtained from a second manufacturer, must be analyzed to verify that the standards used to create the initial calibration curve were prepared correctly.

8.) Write the sequence as below, identifying the type of sample as initial calibration standards, the LIMS identification of the standards, and the applicable dilution factors. The "stype" and S-number must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information and should be written into the sequence as follows:

Diesel Calibration Where: <S#> is the LIMS S# of the standard used (ie: S3124)

IB
CMARKER,S#
IB,CALIB
ICAL,S#,DSL_10
ICAL,S#,DSL_100
ICAL,S#,DSL_500
ICAL,S#,DSL_1000
ICAL,S#,DSL_5000
ICAL,S#,DSL_7500
IB
ICV,S#,DSL_500

9.) Use the same EZchrom method as used for samples.

ACCEPTANCE CRITERIA & DATA REVIEW

After the standards have run, integrate the fuel standards then print the runs to "EZChrom_ capture" printer. Verify that the curve passes acceptance criteria. Any corrections must be done through EZchrom, then resent to LIMS and a new ICAL# created. Any data processed with the draft ICAL would then need to be reprocessed against the corrected, new ICAL#.

10.) See Appendix 9 for instructions on working up the EZChrom and LIMS files.

11.) Verify that each compound was detected, identified, and integrated correctly in each of the standards.

Unsubstantiated alteration of peak integration solely to pass calibration criteria is illegal and is grounds for immediate termination.

12.) Print the files to EZChrom_Capture.

13.) For the alkane (carbon-marker) standard, examine the data closely to make sure that all of the required peaks were detected and labeled correctly.

14.) Print out a hard copy of the EZChrom ICAL curve for each compound.

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15.) Verify that the %RSD is ≤ 20% for each of the common (or needed) carbon-ranges and that the upper levels are not saturated.

16.) If the curve fails the %RSD criterion or the high concentration standards appear to be saturating the detector (decreasing response factors in comparison to the lower level calibration standards), review the results against the following criteria:

- The low point may only be rejected for those compounds that have reporting limits greater than that level.
- The high point may be rejected for compounds that tend to saturate at high levels so long as there are at least 5 points remaining for each compound in the ICAL.
- If a single point in the curve is causing the failure, the standard may be reanalyzed, so long as it immediately follows the original curve and all compounds are calibrated using the second run.
- Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular compound.

17.) Generate the LIMS ICAL summary and verify that the data match the EZChrom report.

18.) Examine the LIMS ICAL summary. The %D for recalculated concentrations should be within 20% of the true concentration of the standard.

19.) Using the newly calibrated method, process the Initial Calibration Verification (ICV) standard, to verify that the calibration standards were prepared correctly and to highlight any discrepancies between the primary- and second-source standards. See Continuing Calibration Verification Section for the procedure to generate this form.

The ICV should meet the CCV criteria of ≤ 15%D.

If the first ICV does not meet the acceptance criteria, another ICV standard may be analyzed; “x” out the first ICV and process the data from the second ICV. Be aware that if the second ICV is processed, that data must be used.

Note: The method 8015D requirement for the ICV & CCV %D is ≤ 20%, however CA-DHS has not yet recognized 8015D, so C&T must continue to meet the 8015B for any client that does not list 8015D in a project-specific QAPP.

20.) Review & sign each data file. Complete the “GC & HPLC ICAL Review Checklist” and notify the Department Manager or QC Chemist that the calibration is ready for review; the ICAL cannot be used to process final forms through LIMS until it has been reviewed and approved in LIMS.

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APPENDIX_9: WORK UP INITIAL CALIBRATION FILES

MULTI-POINT FUEL OR SURROGATE CALIBRATION _____

1.) Update the RT "Carbon-Markers" (Alkane Standard) & create new RT method

Before processing the ICAL files, update the retention times so the correct portion of each chromatogram will be quantitated.

1.1) In EZChrom, open the sequence containing the carbon marker run.

1.2) Open the most recent carbon marker method file.
- -File
  - -Method
    - -Open
      - - (the most recent carbon marker method)

1.3) Save this method under a new method name with the same Julian date as the sequence containing the carbon marker runs (example: carbon markers that ran on Feb.13 should be saved as "cm044.met").
- -File
  - -method
    - - save as...

1.4) In the carbon marker data file, label each peak with its RT by right-clicking, select annotations, then select Retention Times and hit 'Apply'. Each peak should now have the RT labeled at top of each peak.

The identity of the first peak depends on the alkaline mix, and instrument parameters. The current mix includes C8 – C50, but only GC26 can see C8; for all other GC's, the first peak after the solvent peak is C10 (decane), followed by the even numbered alkanes (C12, C14, etc) out to C40, followed by C50.

1.5) If any peaks are not labeled, manual peak integration may be necessary to correctly define the peak. Print a copy of the chromatogram to \\LIMS\EZChrom_capture, then print out a hard copy by going to the sequence where the carbon markers ran, click on the raw data for the carbon markers, then click "Reprint" on the chromatogram file.

1.6) Update the new Carbon Marker (cm) method with the current RT's by opening Method, Peak/Groups, and type in the new retention times.

1.7) Save the method!

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1.8) Reprocess carbon-marker data file using the new carbon marker method, so that the peaks are labeled with the correct names.
-Sequence
-Process
-Range (enter the run number here)
-click on Start

1.9) Print another copy of the data file chromatogram to Ezchrom_capture. This will have each peak named correctly and will overwrite the previous chromatogram that was sent to LIMS.

1.10) Print a copy of the method report to EZchrom_capture by selecting File, Print, Method.

2.) Update the Retention Times

2.1) Open the most recent surrogate method and save this method under a new method name with the same Julian date as the sequence containing the ICAL data files.

2.2) Click on:
-Method
-Peaks/Group
-Named Peaks

2.3) Under the “Ret. Time” column enter the time from C26

2.4) Save the changes that you made to this method.

2.5) Open the most recent TEH method and save this method under a new method name with the same Julian date as the sequence containing the ICAL data files. Click on:
-Method
-Peaks/Group
-Named Peaks

2.6) Under the “Ret. Time” column enter the correct retention times for each alkane.

Click on the “Groups” tab.

Each row has a compound with a carbon range.

For each row, click on the box under the “Group Def.” column.

A “Group Range Definition” box will pop up with a 2x2 chart.

One column will be “Region Start” and the second will be “Region Stop”.

Enter the retention time for the starting alkane in the first column and the retention time

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for the second alkane in the second column.

Click on “OK”, this will close the window.

2.7) Repeat this for every carbon range.

2.8) When finished save method and double check to make sure that all retention times were entered correctly.

2.9) Open each method (surrogate and TEH) and print the method summary, this is done by clicking on:
- File
  - Method
    - Print

3.) Integrate the Fuel Calibration Files

Once the EZChrom methods have been created with the correct RTs, it is time to process the fuel calibration points.

3.1) In the sequence containing the ICAL data files, under the ‘Method’ column, select the method with the updated retention times.

3.2) Process the data files once to draw up the baselines, carefully integrating only when necessary. Do not send any data to LIMS at this time.

3.3) Under the “Level” column, enter the correct calibration level.

To find the calibration level, go to the “Groups” tab where the retention times were entered. In this window, scroll to the right and there are columns labeled “Level 1” through “Level 30”, where the number represents the concentration for that compound (in mg/L).

3.4) In the “Run Type” column, select “Clear Calibration at Level” for each calibration point.

3.5) Reprocess each data file. This will now update the calibration with the new area counts.
CAUTION: If the newest method name is not in the method column, you will overwrite whatever method is in the column!

3.6) Under ‘Method’, ‘Review Calibration’, check to make sure the %RSD for each analyte range being calibrated is $\leq$ 20%.

You may need to change the view mode from peaks to groups (or visa versa) by right clicking in the window on the upper right, select “View Mode,” and then select either the peaks or groups view mode.

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3.7) If the %RSD passes, print out the Peak/Groups report. Do this by left-clicking on a range for the analyte being calibrated in the upper right window, then right-click and select "Print Current Peak/Group." Repeat this for each range being calibrated. These printouts will be necessary to review EZChrom method data and make sure it matches LIMS.

3.8) Save the changes made to current method.

3.9) Print all method report and chromatogram files to EZChrom_Capture.

4.) Create a Calibration in LIMS

4.1) Work up the ICAL in EZChrom, making sure the printers are set to EZChrom_capture and that the calibration blank is labeled "IB,CALIB".

4.2) Review and sign the runs in the LIMS sequence.

4.3) In the LIMS Sequence page, select the runs needed for the ICAL by checking the box next to each file.

4.4) From the task bar below the sequence, click 'Create calibration'.

4.5) Delete any ranges that are not in the CCV list.

4.6) Verify that the raw data matches the calibration table.

4.7) Verify that the response factors in EZChrom match those in LIMS.

4.8) If the calibration is a single-point calibration, verify that the surrogate area was subtracted, if the surrogate falls within the fuel range.

4.9) Sign the sequence and the ICAL itself in LIMS.

4.10) Print the following:
- Carbon markers
- EZChrom method printout (to review that correct RTs were entered)
- EZChrom ICAL printouts
- LIMS ICAL summary

4.11) Sign and date each page and then submit the printed copies to the data reviewers.

4.12) When the reviewers return the hardcopies, file the hardcopy in the data archives.

SINGLE-POINT CALIBRATION _____

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1.) Integrate the CCV to be used for the ICAL using the most recent analytical method.

2.) Print the report using our TEH format.

3.) Determine what carbon range is being reported for the fuel. On the analytical report, highlight the area of the fuel and its carbon range.

4.) If the surrogate (hexacosane, C-26) does fall within the carbon range, subtract the surrogate's area from the area of the fuel.

   a.) On the TEH surrogate report, highlight the area of the surrogate.

   b.) Subtract the area of the surrogate from the area of the fuel.

   c.) Divide that adjusted area by the known concentration of that CCV. The result is the Calibration Factor for the fuel that should be entered into an updated method. Write this calculation on the analytical report along with your initials and the date.

5.) If the surrogate (hexacosane, C-26) does not in fall in the fuel's carbon range, take the area for the fuel and divide it by the known concentration of that CCV. This result is the Response Factor for the fuel that should be entered into an updated method. Write this calculation on the analytical report along with your initials and the date.

6.) Create a new method name, using the Julian date of the sequence containing the CCV, just as you would if you were creating a multi-point calibration.

7.) Enter the fuel's Response Factor into the analytical method.

   a.) Open the new method with the CCV's Julian date.

   b.) In EZChrom click on:
      - Method
         - Peak/groups...
            - Click on the "groups" tab

   c.) Scroll right until you find the "Manual RF" column.

   d.) Enter the response factor that you calculated.

   e.) Save the method.

8.) In the LIMS sequence, change the Sample type of the fuel CCV from CCV to ICAL.

9.) Make sure that the data was printed to the EZChrom_capture printer.

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10.) In the LIMS Sequence page, check the box of the run that is being sinle-pointed. From the task bar below the sequence, click 'Create calibration'.

11.) Print the LIMS calibration summary and make sure that it matches EZChrom.

REVIEW THE CALIBRATION _____

1.) Compare the calibration response factors generated by LIMS to the factors generated by EZChrom. Did your calibration factors change significantly from the last time you calibrated this GC?

2.) Print out the ICV and CCVs with the updated surrogate method and then draw them with the updated analytical method. Process the data in LIMS. Does the EZChrom result match the LIMS result? Is LIMS using the updated calibration factor? You can determine this by checking the Cal. Number on the LIMS report. Did you remember to update the description field in your EZChrom method? Spend some time looking at your calibration before you assemble it.

3.) Once you are very confident that everything is acceptable, get an ICAL review checklist and assemble it in order. Submit it for review. You will only be able to put out draft data until the ICAL is reviewed.

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APPENDIX_10: USING EZCHROM

Set up a new sequence in EZChrom:

1.) Open the latest sequence and rename with current Julian date. To do this:

1.1) From the menu bar, select FILE, SEQUENCE, and OPEN.
1.2) If a sequence is already open, SAVE that sequence then create a new sequence by going to FILE, SEQUENCE, SAVE AS.
1.3) Save sequence under the new Julian date.seq (e.g. January 17 = 017.seq).

2.) Once the new sequence is created, change FILENAME to the current Julian date. In the FILENAME column, select the first box in the beginning of the sequence, right click, and select fill down. Enter Julian date/channel/number bracket (e.g. 017a<###>), then select OK.

3.) Next, make sure that the most current surrogate/run method is open. From the sequence, select the first box under the Method column, and click on the green diamond box. Select the most current surrogate method and OPEN. Right click on the opened method and select 'Fill Down'.

4.) Clear the old SAMPLE ID column, and type in the new sample IDs and CCVs that are going to run.

5.) SAVE new sequence.

Run the Sequence:

6.) Open the sequence that you want to run.

7.) Set GC to run sample(s) by selecting the CONTROL button from the toolbar, and select Sequence Run.

8.) Enter the sequence run numbers for the range you want to run. If you want to run vials 1 through 10, type in 1-10. If you want to run multiple vial ranges, type them in consecutive order, with a comma between each range (for example: 1-10, 21-25).

9.) Click the Submit (or Start) button.

10.) Watch to make sure that the autosampler picks up the intended vial.

Process Surrogate Results:

First process data with the surrogate method.

11.) **Make sure that samples are processed with the most recent surrogate method**. To select

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the most current surrogate method, under the Method column, click on green diamond button, and select the most current method from the menu.

12.) Click on SEQUENCE, and select Process.

13.) Enter the data file range that you want to process and uncheck the 'Print Method Reports' if it is checked. Check 'Review' box and select results review, and hit Start.

14.) No special manual integration should be necessary for surrogates. If the sample contains many alkane peaks, which may coelute with the surrogate or within the surrogate retention window; examine the data closely to ensure that the correct peak was picked and integrated correctly.

15.) Print method custom report under REPORTS, PRINT, METHOD CUSTOM REPORT.

Process the hydrocarbon (TEH) results:
After the data has been processed with the surrogate method, process the hydrocarbon results.

16.) **Make sure that samples are processed with the most recent teh_method**. To select the most current teh_method, under the Method column, click on green diamond button, and select the most current method from the menu.

17.) Click on SEQUENCE, and select Process.

18.) Enter the data file range that you want to process and uncheck the 'Print Method Reports' if it is checked. Check 'Review' box and select results review, and hit Start.

19.) Carefully integrate each sample using the EZChrom integration tools. Print method custom report under REPORTS, PRINT, METHOD CUSTOM REPORT.

20.) Make sure a good chromatogram is printed for each sample by scaling it to the appropriate size, right clicking with the mouse, select Utilities, and Print.

21.) After all samples are processed in EZChrom, review the data in LIMS.

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APPENDIX_11: WORK UP DATA IN LIMS

Work up a sequence in LIMS.

1. Process sequence in EZChrom, making sure EZChrom print setup is set to \\LIMS\ezchrom_capture.

2. Go to your Sequence in LIMS. Select and process runs. In the top right, use the "Review" pull down menu and select your Sequence (for example: GC11A/ 02/11/07).

3. Check each run to ensure it is within the acceptable criteria.

   3.1 Scroll down the left screen and make sure the EZChrom TEH raw data and surrogate raw data reports match LIMS, and check for the proper chromatograms.

   3.2 Use the re-run button to mark data for re-analysis. Any re-runs that are not because of linear range will require a brief comment as to why the data needs re-analysis.

   3.3 For over linear range or over diluted samples, use the RR button with the rerun dropdown tab selected and type in the dilution factor needed. Finally, use the RX button for any re-extracts.

4. Click the “integrated” canned comment, then Save+Next, after the run has been carefully reviewed.

Put together a Batch QC pack in new LIMS

Batch QC packages can be put together after the extraction lab has scanned in all the sample prep paperwork, and all the QC samples (including the MSS and any necessary re-runs) have run and their brackets have been closed off with CCVs.

1. Use the C&T search function and type in batch number. This will find all data associated with the batch. Use the review apps pull-down menu and select batch number (ie: batch 121060).

2. Log in if necessary.

   Important! You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

3. In Review Application mode on left screen, the runs for the QC will appear along with associated ICALs, CCVs, and sequences. The associated CCVs should already be signed. If any are not signed, review and sign them.

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4. Click the batch number at top of screen. Scanned documents should appear on right screen. Scroll down and check for any Corrective Action Records (CAR's) associated with the batch or with samples in the batch. Review and update the CAR.

5. Review the sample prep log to make sure volumes, spike, and surrogate amounts are entered correctly in LIMS (data on left screen). Also look for any comments in the comment section, such as "2x surrogate" or "sediment".

   For soils, make sure the weights in the soil aliquot page are entered correctly in LIMS.

   Finally, be sure the correct prep and clean-up methods are entered.

6. Click on each QC data file and verify that all QC requirements (clean MB, analyte recovery, surrogate recovery, RPD) are met and that any necessary comments explaining recoveries that are outside of QC limits are present.

7. Check that the LIMS raw data numbers match the EZChrom data.

8. Sign each QC file, signifying that the data has been reviewed and is reportable or has been narrated if it is not being reported.

9. Verify that the run of the MSS that you are reporting is the same as the one that is linked to the MS/MSD. If the MSS was re-run for any reason, the run that we are reporting may not be linked correctly. If necessary, change the MSS run that is linked to the MS/MSD using the "MSS" link from the sequence where the MS/MSD ran. It must be changed for both the MS and the MSD individually. Be sure to make comments about any MS/MSD recovery or RPD failure.

10. Add any necessary comments to the batch as a whole using the comment tool at the bottom of the screen. For example, this would be where you could explain that the MS/MSD were not run if the MSS was run at a dilution >5x.

11. Finally, click the "Sign" button when the batch QC pack is complete and ready for review.

12. Write the batch number on our tracking list of batches sent for review, and notify a QC reviewer that it is ready (by instant message, telephone, or in person). Cross the batch number off the list once a reviewer has replied the data is reviewed.

Work up a Job

1. Review job sheet. Check to make sure the correct cleaned-up extract of samples and QC has run and are ready to report. Make sure that any client-specific needs are addressed (for example, CCI needs to be on one instrument, and ITSI Hunter's Point requires special flagging). Also, double-check to make sure the job was logged in properly. Finally, search for any CARS associated with job.

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2. Once you know what you need to report, begin by using the C&T search tool and type in job #, product (gcsv), and matrix (ie: 194505 gcsv soil). This will bring up sample data in LIMS. In the top right, use the "Review" pull down menu and select the job you would like to report.

3. Login if necessary.

   Important! You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

4. Click on first sample. This sample should already have an “integrated” comment by the analyst who integrated the data. The sample chromatogram should be present on the right monitor. If this chromatogram needs to be compared to CCVs for flagging, size the window to fit half the screen. Open a new window in the right monitor and open the CCV chromatogram(s) needed for flagging. Size this chromatogram to fill the other half of the right monitor. This should allow you to view the sample chromatograms along side the CCV chromatograms.

5. Go through each sample and choose ("u"), flag, and sign each reportable run. If there are multiple runs for one sample, you can click on the “chooser…” button located below sample results to view all runs on one screen. If there are only two runs to compare, use the “compare next” button to compare and select which run(s) to report.

6. Once all specific versions (and general versions) of the QC and samples have been chosen, flagged, and signed, select the pull down menu that says "all" and select "pkg". While in package mode, select “edit runs” and look at the flag by individual analyte to check for consistency. Click “Done” to return to previous screen.

7. Click ‘Reports’ then choose which Form 1's to print. Print those forms on “2nd page” letterhead.

8. Next click “Done”. The Form 1's will now appear on the right in the Review App.

9. If the job is being turned in as a F1 without closing CCV's or without the QC package completed, turn in F1's with job sheet to reviewers at this time. If entire package is complete, and CCV’s have been analyzed, go on to next step.

10. Click on Checklist to generate the checklist and print it out. Hit Done.

11. Make any necessary comments for the Job.

12. Click Sign to sign off on the Job. Remember to log out by clicking on your initials.

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Curtis & Tompkins, Ltd

POLYCHLORINATED BIPHENYLS (PCBs)

EPA 8082 & EPA 608

Approved by:	Signature:	Date:
Cheryl Bowden Operations Manager	Cheryl Bowden	4/18/13
Teresa Morrison QA Director	Teresa Morrison	4/11/13
Reapproved:		

This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

Curtis & Tompkins, Ltd

Polychlorinated Biphenyls (PCBs)
EPA 8082 & EPA 608
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This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

Curtis & Tompkins, Ltd

POLYCHLORINATED BIPHENYLS (PCB's)

1.0 SCOPE

This document describes the analysis and quantitation of PCB's that have been extracted from liquid samples, solid matrices, and wipes or other miscellaneous matrices. The analysis is performed by first injecting the hexane extract into a capillary column GC to separate the components into discreet compounds. An Electron Capture Detector detects those components that are halogenated compounds and the analyst uses pattern recognition to identify the Aroclor mixtures. Analysis for the quantitation of the discreet PCB congeners is described in a separate procedure.

Sample concentrations are reported in micrograms per liter (µg/L) for water samples or micrograms per kilogram (µg/Kg) for solid samples. See Appendix 8 for the compound list and reporting limits.

EPA 8082 was written by the EPA's Office of Solid Waste with additional guidance for surface water and ground water, as opposed to EPA 608 which was written by the EPA's Office of Water specifically for wastewater. EPA 608 may also be requested for groundwater samples if the client is planning to discharge the water, with or without additional treatment, into a wastewater stream or into naturally occurring surface waters (bay or river). See Appendix 13 for EPA 608 requirements.

2.0 REFERENCES

Analytical Methods:
EPA 8082, Polychlorinated Biphenyls (PCB's), SW-846, Dec 1996
EPA 8082A, Polychlorinated Biphenyls (PCB's), SW-846, Feb 2007
EPA 8000B, Determinative Chromatographic Separations, SW-846, Dec 1996
EPA 8000C, Determinative Chromatographic Separations, SW-846, Mar 2003
EPA 608, Organochlorine Pesticides & PCBs, 40CFR136 Appendix A

Extraction & Cleanup Methods:
EPA 3500B, Organic Extraction and Sample Preparation, SW-846, Dec 1996
EPA 3500C, Organic Extraction & Sample Preparation, SW-846, Feb 2007
EPA 3510C, Separatory Funnel Liquid-Liquid Extraction, SW-846, Dec 1996
EPA 3520C, Continuous Liquid-Liquid Extraction, SW-846, Dec 1996
EPA 3540C, Soxhlet Extraction, SW-846, Dec 1996
EPA 3550B, Ultrasonic Extraction, SW-846, Dec 1996
EPA 3550C, Ultrasonic Extraction, SW-846 Feb 2007
EPA 3660B, Sulfur Cleanup, SW-846, Dec 1996
EPA 3665, Sulfuric Acid Cleanup, SW-846, Dec 1996
EPA 3580A, Waste Dilution, SW-846, Dec 1996
EPA 608, Organochlorine Pesticides & PCBs, 40CFR136 Appendix A

Related References:
C&T SOP SVOC 3.0, Electron Capture Detectors
C&T SOP QA 1.4, Balance Calibration Check & Maintenance
C&T SOP QA 1.5, Calibrating & Maintaining Temperature Controls

C&T SOP QA 1.6, Pipette Calibration Check Procedures
C&T SOP QA 4.1, Establishing Control Limits
C&T SOP QA 4.4, Determining Method Detection Limits (MDL)
C&T SOP QA 4.6, Limit of Quantitation (LOQ)
C&T SOP QA 9.6, Insuring Compliant Manual Integrations
TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Rev 4.2 October 2010
DoD Quality Systems Manual (QSM) Version 5/ DoE QSM Version 3, Final Draft, Mar 2013

3.0 PRESERVATION & HOLDING TIME

Preservation:	No chemical preservation.	
	Store samples and extracts at > 0.0 to 6.0^{o} C	
Holding time:	H_2O:	7 days from collection to extraction*.
	Soil:	14 days from collection to extraction.
	Extract:	40 days after extraction.

* *EPA 608 Method Note:* EPA 608 requires that if the extraction is not begun within 72 hours of sample collection, the sample pH be adjusted to 5.0 – 9.0 SU with NaOH or H_2SO_4 to extend the holding time to 7 days.

4.0 SAFETY

Assume that all samples contain hazardous and/ or potentially toxic chemicals and should be handled with care. Safety glasses, gloves, and a lab coat should be worn whenever handling samples, extracts, reagents, or standards.

5.0 QC REQUIREMENTS

A method blank (MB), laboratory control sample (LCS), matrix spike (MS) and matrix spike duplicate (MSD) are extracted and analyzed with every batch of twenty or fewer samples. If insufficient sample was submitted to perform matrix spikes, a blank spike (BS) and blank spike duplicate (BSD) will be extracted and analyzed in place of the LCS/ MS/ MSD. Two surrogate compounds (TCMX & DCB) are added to each sample, method blank and spike to monitor the performance of the extraction and analysis, and to each standard to verify that the extract was injected correctly.

An initial calibration curve is generated, using a minimum of five points, for Aroclor 1016 and 1260; if a quadratic curve is used, it must include 6 points at minimum. A single point for all other Aroclors is analyzed for pattern recognition. If using Average Response, the RSD for the ICAL curve must be $\leq$ 20%; if using linear regression, the curve must meet a correlation coefficient r $\geq$ 0.995 ($r^2 \geq 0.990$).

If one of the other Aroclors is identified, the calibration verification standard immediately preceding the sample can be used to used to establish the calibration factor for that Aroclor, so long as the 1016/1260 ICAL has demonstrated the linearity of the detector (as per 8082 Section 5.6.2).

DoD QSM: For projects associated with the DoD QSM, a minimum of five points is required for each Aroclor and surrogate.

A mid-level initial calibration verification (ICV) standard obtained from a second supplier is analyzed immediately following the calibration curve. For projects requiring EPA 608 or 8082; the ICV acceptance criterion is %D ≤ 15%. For projects referencing EPA 8082A or the DoD QSM, the ICV acceptance criterion is %D ≤ 20%.

A continuing calibration verification (CCV) of Aroclor 1016/1260 standard is analyzed at the beginning of each sequence, after each group of 10 field samples, and at the end of the sequence. The concentration of the CCV standard should be alternated within the calibration range, excluding the highest and lowest points. For projects requiring EPA 608 or 8082; the CCV acceptance criterion is %D ≤ 15%. For projects referencing EPA 8082A or the DoD QSM, the CCV acceptance criterion is %D ≤ 20%. If the %D is outside this limit, use the guidance in the Data Review section below to determine the appropriate corrective action. If other Aroclors are detected, the extracts should be rerun bracketed by those Aroclors.

The Batch QC Acceptance limits for PCB's are generated semi-annually, through control charts of the previous two years batch QC data. A method detection limit (MDL) study is performed whenever a new instrument is installed or significant changes are made to the extraction or analysis procedure. Limit of Detection (LOD) and Limit of Quantitation (LOQ) samples are analyzed quarterly. See the associated QA SOPs for details.

EPA 608 Method Notes: Method EPA 608 includes somewhat different requirements and acceptance limits; see Appendix 13 for discussion of these requirements.

Note: For samples associated with site-specific project plans developed by the client, the requirements of those plans supersede the requirements listed in this document.

6.0 EQUIPMENT (see Appendix 4 for instrument specifications & conditions)

Hewlett-Packard Gas Chromatographs:

GC:	Hewlett-Packard Model 5890
Detector:	Hewlett-Packard Model 19233 or G1223A, Electron Capture Detector
Autosampler:	Hewlett-Packard Model 7673A or 767B)

Varian Gas Chromatographs:

GC:	Varian Model CP-8400
Detector:	Varian 02-001972-00, Electron Capture Detectors:
Autosampler:	Varian Model CP-8400

Columns:
The following columns are typically used because they demonstrate superior separation of the PCB congeners. Others are available and may be used for the Aroclors analysis, so long as the pertinent information is documented in the maintenance log.

Front (Channel A): Restek Rtx-CLP Pesticides, 30m x 0.32-mm I.D., 0.5 µm df
Back (Channel B): Restek Rtx-5, 30m x 0.32-mm I.D., 0.5 µm df

EZChrom data acquisition and processing software

7.0 ROUTINE MAINTENANCE

Generally, the septum, injection liner, and blue seal should be changed every couple of days, before analysis of an opening Continuing Calibration Verification (CCV). The injection liner is a 4mm splitless single goose-neck liner. The column may need to be trimmed (usually a minimum of 15cm) if oily or highly contaminated samples were analyzed in the previous sequence; additional trimming of the column may be necessary, depending on the results of the analysis of the CCV standard. See Appendix 5 for additional maintenance procedures & details.

8.0 DAILY SEQUENCE

Each sequence should begin with an instrument blank followed by a Continuing Calibration Verification (CCV) standard. Once CCV has passed acceptance criteria (see Appendix 10 for criteria), sample extracts may be added to the instrument sequence. Additional instrument blanks and CCV's must be analyzed after every ten field samples, not including known batch QC samples, and at the end of the sequence. The concentration of the CCV's should be varied within the calibration range, excluding the highest or lowest points. Instrument blanks may be included after each CCV bracket to demonstrate that instrument contamination is not contributing to the reported results.

All solutions are analyzed on both columns in the following sequence:

Hexane Blank
CCV – Ar1016/1260
Hexane Blank (optional)
Method Blank
Blank Spike
Blank Spike Duplicate
10 Samples
CCV – Ar1016/1260
Hexane Blank (optional)
Up to 10 Samples
CCV – Ar1016/1260
Hexane Blank (optional)
Up to 10 Samples
CCV

The sequence must end with CCV regardless of the number of samples analyzed.

Although the current SW-846 methods allow up to twenty runs between CCV's, C&T typically runs CCV's after every ten samples to meet the additional SW-846 requirement that no more than 12 hours should elapse between CCV's and to reduce the number of

reanalyses caused by failing CCV's. Additional samples may be analyzed between CCV's so long as the 12-hour window is met.

If the instrument will be running unattended or overnight, it is a good idea to load two CCV standards for each bracket, to reduce the number of samples that have to be reanalyzed due to an injection error.

DoD QSM: The DoD QSM requires CCVs to be run after every 10 field samples (excludes batch QC and instrument standards/blanks). If the sequence includes DoD QSM 4.2 samples, the CCVs immediately bracketing those samples must pass the QSM criteria.

EPA 608: EPA 608 only requires that a CCV be analyzed once daily, however C&T standard practice is to follow SW-846 guidelines as described above.

8.1 File Naming Conventions

Various user reports are automatically produced after the run is complete. This automation is based on the type of sample being analyzed, the sample number, the LIMS identification of any associated calibration or spiking standards, the batch number, and any applicable dilution factors. This data must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information and should be written into the sequence as follows:
Date files are named using the Julian date, followed by a dash, and then the run number (e.g. 123-002). These files are written to the G:\ezchrom\Projects\GCxx\Data subdirectory, where xx is the GC number.

Examples of EZChrom method (processing) file names include (where ### is the Julian date):

Aroclor 1016 / Aroclor 1260:	pcb-run-###.met
Aroclor 1221 / Aroclor 1254:	pcb-ar2154-###.met
Aroclor 1248:	pcb-ar1248-###.met

Examples of other types of file names and paths include:

Data files:	G:\ezchrom\Projects\GC16\Data\219-015
Sequence files:	G:\ezchrom\Projects\GC16\Sequence\219.seq
Report format:	G:\ezchrom\Projects\GC16\Template\Ar1254-REPORT-04-13-12.rep

See Appendix 11: Using EZChrom for additional instructions for writing the sequence into the software.

8.2 CCV (Continuing Calibration Verification) (See Appendix 10 for Acceptance Criteria):

Begin each sequence with an Aroclor 1016/ Aroclor 1260 CCV ("AR1660" Continuing Calibration Verification) standard to verify that the response of the instrument has not changed significantly and that the curve may still be used to quantitate sample results. Use a standard at one of the mid-levels of the calibration curve; do not use either the highest or

lowest point. It is also a good idea to run Aroclor 1254 with samples from a new site, since this compound is also fairly common.

EPA 608 Method Note: EPA 608 only requires analysis of a daily CCV, however C&T standard practice is to follow SW-846 guidance and analyze a CCV at the beginning of the sequence, every 12 hours, and at the end of the analytical sequence.

8.2.1 Decide what CCV standards to analyze, based on the spiking compound used in the batches to be analyzed and keeping in mind that the concentrations should be alternated across the mid-levels of the calibration.

Note: The USACE recommends that the ICAL standards be used as CCV's, in order more readily determine which problems are due to changing instrument conditions and are not due to differences between standards.

8.2.2 Load CCV standards after every ten samples and at the end of the sequence, excluding batch QC, instrument blanks and other standards in the count. Remember to add any other Aroclor CCVs bracketing extracts that are known to contain non-1016/1260 Aroclors or in which these other Aroclors have been identified.

LIMs identifies samples that are associated with the DoD QSM requirements, other DoD requirements or commercial clients. It identifies all project specific criteria that are reviewed by the analyst before sample analysis.

For sequences containing samples that are not associated with requirements from DoD QSM, if the instrument is running unattended or overnight, it is a good idea to load two CCV standards for each bracket, to reduce the number of samples that have to be reanalyzed due to an injection error. Type in the sequence with an "x" stype for the second CCV so that only the first CCV will automatically process.

If the sequence includes DoD QSM 4.2 samples, the CCVs immediately bracketing those samples must pass the QSM criteria.

8.2.3 Analyze the standards using the same data acquisition method as for the samples, typing "CCV," before the working standard number, so that LIMS will automatically generate and print a CCV summary, which compares the calculated concentrations from this run to the known concentrations of the standard.

8.2.4 Examine the CCV summary against the criteria listed in Appendix 10 to determine whether the CCV is acceptable.

8.2.5 If the acceptance criteria are not met, examine the integration to verify that the peaks were correctly integrated. Manual integrations must be consistently applied to standards and samples. If manual integrations or baseline corrections are performed, resend the file to LIMS and generate a new CCV summary.

Unsubstantiated alteration of peak integration solely to pass calibration or QC criteria is illegal and is grounds for immediate termination.

8.2.6 If the acceptance criteria are not met, analyze another CCV standard. If the second analysis of the standard fails to meet the criteria, recalibrate and/or perform other instrument maintenance.

8.2.7 If the CCV's fail acceptance criteria, data may be reportable based on the following criteria:

a. If the failing compound is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b. If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c. If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.

d. If the compound fails the %D criterion due to a low response and *was* detected (even below the reporting limit), the sample must be reanalyzed.

8.2.8 Load additional CCV's after every ten samples and at the end of the sequence. The standard concentration used for the CCV should be alternated over the course of the sequence. See Appendix 1 for calculation of %D.

8.3 Prepare the Sample and Batch QC Extracts for analysis

8.3.1 Remove the extracts from the extraction lab refrigerator and let the extracts warm to room temperature then aliquot approximately 1mL of extract into an autosampler vial.

Note: If the extract is dark & oily or opaque, do another acid cleanup on the extract. If the extract is still highly colored, make a dilution that will result in an extract that is light yellow in color. If dark, oily, or viscous extracts are analyzed, a tarry residue will build up in the injection port, causing active sites and failing CCV's.

8.3.2 If dilutions are required, see Appendix 3 for instructions on preparing the dilutions.

8.3.3 Place the samples on the autosampler tray beginning with the lightest colored extracts followed by more highly colored or viscous extracts.

9.0 QUALITATIVE ANALYSIS

Each Aroclor is a mixture of many individual polychlorinated biphenyls (for example: 3,3',4,4'-tetrachlorobiphenyl is a single component "congener" of an Aroclor mixture). Aroclor identification is based on pattern recognition, through comparison of the sample pattern to those of the Aroclors used as calibration standards. Trace overlays on EZChrom are very useful in determining which, if any, Aroclors are present because it can be used to overlay the sample chromatogram with the standards chromatograms.

Note: Although second-column confirmation is not required by EPA 8082, as the identity is based on pattern recognition and not on an individual peak, most of C&T's Department of Defense (Army Corp, Navy, AFCEE) clients do require it. C&T standard practice is to work up data from both columns and report the higher of the two results, unless specifically directed otherwise by a client's project plan.

9.1 Multi-component Pattern Recognition

Aroclors are not discrete chemicals but are comprised of many related Polychlorinated Biphenyl (PCB) compounds. For environmental samples, these analytes are identified by comparison of the sample chromatograms to those of the standards. The composition and relative ratios of the target peaks in the sample chromatogram should *resemble* the standard but do not have to exactly match that of the standard, since the composition of these analytes is not completely defined, may vary between manufacturers and from batch to batch, and may have changed as the sample is "weathered" in the environment.

Except for Aroclor 1016 and Aroclor 1260, the components of different Aroclors often overlap, thus requiring separate calibration standards for these multi-component analytes. If the peaks do not overlap the standards may be combined, as in Ar1016/1260 and Ar1221/1254. For each of the Aroclors, five characteristic peaks are chosen as representative of each analyte; these peaks should be the largest peaks present that do not coelute with other target analytes. To positively identify the analyte in the sample, the chromatogram must contain at least 3 of the characteristic peaks (if present at low levels) with increasing resemblance to the standard as the concentration increases.

Non-target chlorinated compounds such as DDT and Endrin are typically removed prior to analysis by the sulfuric acid cleanup, however Technical Chlordane is not degraded by the acid and may appear in the latter ½ of the run; the two peaks with the highest response are typically a-Chlordane and g-Chlordane. Toxaphene may also be present and elute in the latter ½ of the run.

Use EZChrom's "trace overlay" feature to compare the sample pattern to each of the Aroclor patterns. If an Aroclor other than Ar1016 or Ar1260 is present and CCV's for that Aroclor do not bracket the sample extract, the extract must be reanalyzed with that Aroclor. See Appendix_7 for example chromatograms.

9.2 Peak Identification

Identification of the discrete components of the Aroclors is based on comparison of the peak retention times in the sample to the retention times of the peaks in the mid-level initial

calibration standard or, for single-point calibrations, the retention times of the standard used for initial calibration. For the standards, each peak must fall within its retention time window and be automatically identified on both columns by the data system.

9.3 Integration

Check the integration of the peaks as you examine the chromatogram. Peaks should be integrated from baseline to baseline unless there are obvious matrix interferences such as coelution. For problematic analytes, manual integrations must be consistently applied to ICAL, CCV, and sample integrations.

Determine whether manual integration is necessary by examining the sample chromatogram. For samples in which no matrix interferences are present, the sample peaks should be integrated in the same fashion as the calibration standards. For samples in which interferences raise the baseline, integration of the target compounds should be done on a valley-to-valley basis, unless a nearby negative peak would contribute a positive bias to the reported result; if a negative peak is present, use a baseline event to extend the baseline horizontally across the dip. If manual integration is necessary there are a number of different baseline events to choose from. See Appendix 11 for a listing of EZChrom integration events.

Warning: Unsubstantiated alteration of peak integrations solely to pass QC criteria (ie: calibration, surrogate) is ***illegal*** and is grounds for immediate termination of employment.

10.0 QUANTITATIVE ANALYSIS

Because the components of the various Aroclors overlap and multiple Aroclors may be present in the samples, the entire area of the standard can not be used for quantitation as is done for TPH-diesel (EPA 8015) samples. Instead, five characteristic peaks are identified in each Aroclor; these peaks are used both to assist in pattern recognition and in sample quantitation. A maximum of two of the five peaks may be discarded if it is evident that matrix interferences or “weathering” are distorting the quantitation of the peaks relative to the others.

Analyte quantitation is done using the external standard technique. Quantitation is based on comparison of the area of the target peak to the initial calibration curve for that peak, with adjustments for the sample preparation concentration factor and instrument dilution factor. The concentrations of the selected peaks within each target compound are then averaged to obtain the reported concentration. See Appendix 1 for example calculations. Concentrations are expressed as micrograms per liter or kilogram (µg/L, µg/kg).

All results are reported on a wet-weight (“as received”) basis unless otherwise requested by the client. If the client requests ‘dry-weight’ corrections, the ‘wet-weight’ results in the results database are corrected for moisture by LIMS when producing the final report forms.

10.1 Evaluate the Sample Results

A user report for Ar1016/1260 will be automatically generated once the run is complete. Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

10.1.1 Verify that the target compounds are correctly integrated and bracketed by the applicable Aroclor standards.

10.1.2 If an Aroclor other than Ar1016 or Ar1260 is present and appropriate CCV's bracket the extract, process the data with the appropriate EZChrom method for that Aroclor; see Appendix 11 for EZChrom instructions.

10.1.3 If EZChrom identifies Aroclor peaks but the pattern does not resemble that standard, check the "FP" (false positive) box in the LIMS user page then click "Update LIMS" to save the change.

10.1.4 If the concentration of any of the 5 Aroclor peaks in the sample exceeds that of the highest concentration standard used in the ICAL curve for that compound (or of the highest Ar1016/Ar1260 calibration standard), LIMS will flag that over-range peak. Dilute the extract and reanalyze. See the "Dilutions" section below for further details.

10.1.5 If the Aroclor identified in the sample (see the 'Qualitative Analysis' section above) was different than the bracketing CCV standards, the extract must be reanalyzed with bracketing CCV's of the identified Aroclor. Quantitation is then calculated by averaging the concentrations of three to five characteristic peaks within the PCB pattern.

10.1.6 Neither EPA 608 or EPA 8082 require second-column confirmation for PCBs, however some clients may. If second-column confirmation was required by the client, determine if a result should be reported by reviewing the data from both columns. To be reported, the analyte must be detected on both channels. A tentatively identified hit is considered a false-positive and reported as 'ND' if:

a. a peak is present on one column but not on the other,

b. the peak on the confirmation column falls outside the Rt-window,

c. the result on the quantitation column is ≥2x the reporting limit but less than the reporting limit on the confirmation column, or

d. the result on the quantitation column is <2x the reporting limit but less than ½ the reporting limit on the confirmation column.

If an analyte is detected on both columns with an RPD of ≤ 40% between the two results, report the higher of the two concentrations.

If the RPD > 40% between the two columns, evaluate the chromatograms for any co-eluting contaminants that may be causing the high RPD.

e. If coelution is evident on one chromatogram, report the result from the other column or clean up the extract; narrate the coelution, and the fact that the lower result was reported, on the "Data Review Checklist".

f. If no coelution is evident, report the higher of the two results.

g. LIMS will apply a 'C'-flag to the reported result.

Report the higher of the two columns' results, unless specifically directed to do otherwise by a client's project plan. *Note:* If reporting the lower result, note this and the project name on the Data Review Checklist.

Note: J-flagged hits on primary column will be confirmed by J-flag to MDL on confirm column.

10.2 Interferences & Dilutions

The ECD detector is a highly selective detector for halogenated compounds, which means that although the ECD can detect very low levels of chlorinated compounds in sample extracts, there may also be matrix effects upon the system that are not apparent in the sample chromatogram. Most hydrocarbon and many pesticide interferences are removed during the extraction procedure, when the extracts are treated with sulfuric acid, however the sulfuric acid does not reduce interferences from technical chlordane. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting peaks. Sulfur contamination is often found in sediment samples and can be removed by treating the extract with copper powder, as described in EPA 3660. See Appendix 7 for example sulfur and chlordane chromatograms.

If the extract is dark, oily & viscous, or opaque even after cleanup, repeat the cleanup or make a dilution that will result in an extract that is a very light yellow in color. If black, oily, or viscous extracts are analyzed, a tarry residue will build up in the injection port, causing active sites and CCV failure. See Appendix 3 for preparing various dilutions.

If obvious chromatographic interferences are causing the quantitated result for one or two of the characteristic five peaks to be mis-quantitated, drop that/those peak(s) from the selected peaks so that the reported result is not artificially biased. Keep in mind that there must be a minimum of three peaks used for quantitation of each Aroclor.

If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is

there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

10.3 Surrogates

Surrogate compounds are chemically similar to the target analytes and are added, prior to extraction, to every sample, method blank, and spike to monitor the efficiency of the extraction for that sample. In-house Surrogate Acceptance Criteria are specified in the associated SOP '*8082 Laboratory Control Limits, Table-1*'. These limits are generated semi-annually, using control charts.

After each sample is analyzed, LIMS will automatically generate a user report with the surrogate criteria for that sample and flag any failing recoveries. Evaluate the surrogate recoveries for all samples, method blanks, and spikes. If the the extract was diluted by a factor of 10 or more, the surrogate is considered diluted out and LIMS will place a "DO" flag on the user report and final forms.

If a surrogate recovery is outside QC limits, verify that the prep information (LIMS WS#, amount, and concentration of surrogate added, sample weight/ volume, extract volume, and instrument dilution factors) is correct. If any of these are incorrect, fix the entry and reprocess the data. If the prep entry was correct, determine whether reanalysis is required using the following criteria:

a. If a high recovery is observed but no target analytes were detected above the reporting limit in the sample, note the failure on the 'Data Review Checklist' and report the data without reanalysis, since the possible high bias will not affect sample results.

b. If a high recovery is observed, and target analytes were detected, and there is no obvious chromatographic interference, the sample must be reanalyzed. If the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. If the same surrogate fails criteria after re-extraction it is deemed to be matrix effect. Check the "required for confirmation" box in the LIMS "Review App" for the second batch so it will be included if a Level 4 report is required (or requested at a later date) and note the situation in the case narrative.

c. If a low recovery is observed for any surrogate and there is no obvious chromatographic interference, or documented historical site matrix interference, the sample must be reanalyzed. If the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. Check the "required for confirmation" box in the LIMS "Review App" for the second batch so it will be included if a Level 4 report is required (or requested at a later date) and note the situation in the case narrative.

If a sample must be re-extracted and the holding time has expired, the client's Project Manager must log the sample into LIMS as an alias and have the sample re-extracted as

the new sample number. If the sample is still within holding time, the sample should be re-extracted under the original sample number.

If upon re-extraction, the surrogate recovery is again outside limits, note the matrix effect as "confirmed matrix interference" on the User Report and the Data Review Checklist, otherwise report the data with passing surrogate recovery.

Project-specific quality assurance project plans (QAPPs) may require batch control based on different compounds and control limits, in which case the project requirements supersede this SOP for all samples related to that project.

EPA 608: Method 608 does not discuss use of surrogate compounds, however C&T standard practice is to follow SW-846 guidance and use surrogates as a means of verifying the efficiency of the extraction and analysis.

10.4 BATCH QC RESULTS

For every batch of 20 samples (or less) analyzed, a Method Blank (MB), a Laboratory Control Sample (LCS), a matrix spike (MS) and duplicate (MSD) are extracted and analyzed. If insufficient sample volume was submitted for matrix QC, a blank spike (BS) and blank spike duplicate (BSD) are extracted in place of the LCS/ MS/ MSD.

EPA 608: Method 608 requires an LCS for every 10 samples, so any batch with more than 10 water samples must include a BS and BSD. Matrix spikes are also required on 10% of samples submitted for 608 analysis; if the client does not supply sufficient sample volume for the matrix spike, note this on the case narrative.

Project-specific quality assurance project plans (QAPP's) may contain different requirements than those listed in this SOP. If so, the QAPP requirements supersede this SOP for all samples related to that project.

10.4.1 Method blank (MB):

A method blank is extracted with each batch of samples to verify that the extraction reagents and process are not contributing to the sample results. No compounds should be detected in the method blank, however if a compound(s) is detected, the following steps are used to determine the corrective action required:

a. If the concentration of the contaminant is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

b. If the target compound(s) found in the method blank was not detected in the associated samples, the data may be reported and the problem narrated.

c. If the target compound(s) found in the method blank was also detected in the associated samples, but the level in the samples is greater than 20x the level

in the method blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

d. If the target compounds detected in the method blank were also detected in the associated samples, but at levels less than 20x the level in the method blank, and reanalysis confirms the problem, the samples containing the contaminant must be re-extracted and reanalyzed. Initiate a Corrective Action Report (CAR) immediately so that re-extraction can begin within the extraction holding time, if necessary.

DoD QSM: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), if the sample result for that compound(s) is greater than ten (10) times the amount found in the method blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis. If the sample result for that compound(s) is greater than the reporting limit but less than ten (10) times the amount found in the associated method blank, the samples must be re-extracted and reanalyzed.

10.4.2 **Laboratory Control Sample (LCS) or Blank Spike/ Blank Spike Duplicate (BS/BSD):** Laboratory Control Samples are extracted with each batch of samples to demonstrate the performance of the extraction and analysis in the absence of matrix interferences. In-house Acceptance Criteria are specified in the associated SOP '*8082 Laboratory Control Limits, Table-1*'. These limits are generated semi-annually, using control charts.

EPA 608: Method 608 requires an LCS for every 10 samples, so any batch with more than 10 water samples must include a BS and BSD. EPA 608 defines the batch QC acceptance limits in Table 3 of the method; see Appendix 13 for these limits.

After the QC samples for the batch have run, check the batch QC status to verify that the QC has passed acceptance criteria for all of the client-specified limits associated with the batch; this report will compare the recoveries (and RPD for BS/BSDs) for each compound to the tightest limits applicable to the jobs in that batch. To run this report:

10.4.2.1 Open the LIMS web browser and go to the Pesticide/PCB Instrument Page.

10.4.2.2 Type the Batch number in the "View QC status for batch" box and click VIEW.

10.4.2.3 Review the recoveries, and RPD if applicable, to determine if the data can be reported. If either recovery or the RPD fail acceptance criteria,

reanalyze the QC extracts. If the failure is confirmed upon reanalysis, initiate a Corrective Action Record and use the following criteria to determine the required corrective action:

a. If a high recovery is observed but no PCBs were detected in the associated samples, note the failure on the Data Review Checklist and report the data without re-extraction, as the potential high bias does not affect the sample results.

b. If a high recovery is observed and the samples contain PCBs at levels above the reporting limits, the samples must be re-extracted.

c. If a high RPD is observed but the recoveries are within acceptance limits and PCBs were not detected in the samples at levels above the reporting limits, note the failure on the Data Review Checklist and report the data without re-extraction, as the lack of good precision data does not affect ND samples.

d. If a high RPD is observed and PCB's were detected in the samples at levels above the reporting limits, all samples containing PCB's must be re-extracted.

e. If low recoveries are observed, the associated samples must be re-extracted.

10.4.2.4 If a sample must be re-extracted and the holding time has expired, the client's Project Manager should log the sample in as an alias and have the samples re-extracted as the new sample number. If the sample is still within holding time, re-extract and reanalyze the sample under the original sample number.

10.4.3 Matrix Spike/ Matrix Spike Duplicate (MS/MSD):

Matrix spikes are extracted with each batch of samples to demonstrate the accuracy (recovery) and precision (RPD) of the analysis in real-world samples. In-house Acceptance Criteria are specified in the associated SOP '*8082 Laboratory Control Limits, Table-1*'. These limits are generated semi-annually, using control charts.

EPA 608: Method 608 a matrix spike on 10% of the samples received, however most C&T client submit insufficient sample volume for matrix spikes to be performed. If this is the case, add an "insufficient sample volume submitted" to the case narrative. EPA 608 also defines the batch QC acceptance limits in Table 3 of the method; see Appendix_13 for these limits.

a. If the concentration of a target analyte is greater than 4x the spiking level, LIMS will apply a “NM” (for “Not Meaningful”) flag to those recoveries. Report the data without reanalysis.

   Note: If the concentration of a target analyte is greater than the spiking level, LIMS will flag and footnote that concentration for the client’s attention.

b. If the concentration of a target analyte in the sample is greater than the linear range, but is not greater than 4x the spiking level, dilute the sample, MS, and MSD and reanalyze the extracts.

c. If the concentration of a target analyte in the sample is within linear range but the concentration in the matrix spikes is just greater than the linear range, dilute the sample, MS, and MSD and reanalyze the extracts.

d. If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

e. If the recoveries fail due to obvious chromatographic interference (ie: coelution of other analytes with the spike compounds), narrate the failure on the Data Review Checklist and report the data without reanalysis.

f. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

After the batch QC samples have been reviewed and deemed acceptable, sing the LIMS “Review App” files and submit the forms to the Department Manager or a QC Chemist for review and approval.

11.0 DOCUMENTATION & PEER REVIEW

11.1 Review the job sheet to make sure that any special client needs are addressed (for example, CCI needs to be on one instrument). Make sure the correct clean-up version of samples and QC has run and are ready to report. Finally, search for any CARs associated with job.

11.2 Once you know what you need to report, use the C&T search tool and type in the Job#, Product (“pcb”), and Matrix. This will bring up sample data in LIMS. In the top right corner, use the "Review" pull down menu and select the job you would like to report. If you are unsure how to use the LIMS “Review App”, see Appendix 12 for detailed instructions.

11.3 Log in if necessary.

Important! You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

11.4 Review the LIMS generated user reports, the data reduction quantitation reports, and the chromatograms to ensure that the correct qualifier flags, dilution factors and results are reported. The user report must be initialed and dated by the analyst reviewing and approving the data for that sample.

11.5 Complete and sign the "Data Package Review and Narrative" checklist. The completed data package consists of this checklist, C&T Job sheet, and LIMS “Form 1’s” (sample and batch QC results) and any associated Corrective Action Reports.

11.6 Submit the data package to the Group Leader, Department Manager, or QC Chemist for second-party review. Any changes made by the second-party reviewer must be individually initialed and dated by the reviewer. The second party reviewer must initial and date each user report, make any additional comments on the case narrative and initial and date the completed checklist.

12.0 POLLUTION PREVENTION

Direct the split vent and septum purge lines through a carbon trap in order to reduce solvent emissions into the laboratory. Prepare only sufficient standard volume to use within the shelf-life of the standard to reduce the volume of waste generated by the laboratory.

13.0 WASTE DISPOSAL

All sample extracts should be stored in the Delfield refrigerator in the extraction lab. The extracts should be retained for a minimum of 40 days after extraction. After 40 days they should be included in the ‘PCB’ waste stream.

14.0 REVISION HISTORY

The previous document (revision 9) was updated as follows:

- Cover page and numbering were reformatted
- Section 2: Updated to reflect current guidance documents. Updated all QSM V4.1 references throughout SOP to simply QSM as this SOP is consistent with both V4.2 and V5.0-Final Draft
- Section 3: Storage temperature updated to match current 40CFR and TNI/NELAP
- Section 5: Updated CCV frequency to include only field samples. Reworded to clarify ICV/CCV acceptance limits, based on the method and guidance document. Updated MDL frequency to ‘as needed’.
- Section 8: Replaced ‘CCV level must be varied’ requirement with ‘CCV level should be varied’ recommendation, as this requirement has been dropped from the NELAP standard.
- Appendix_2: Updated standards to match current practice.
- Appendix_4: Added GC-25 settings to Instrument Conditions
- Appendix_8: Removed info for EPA 3545 prep since no longer used. Added Ar1262 and Ar1268 reporting limits.

APPENDIX_1: CALCULATIONS

SAMPLE CONCENTRATION

Quantitation is done by the data system using an external standard calculation method. The concentration of an analyte is calculated, by the data system, by comparing its peak area in the sample chromatogram against the initial calibration curve. This concentration is then adjusted for the dilution/concentration factor to obtain the final concentration of the analyte in the sample.

Note: If a client requests results reported on a 'Dry Weight' basis, the concentration is divided by the 'solids', where the solids is (100-%moisture)/100.

Dry Weight Concentration (ug/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

Concentration using Average Calibration Factor (by external standard)

$$Cx = [\sum (Ax / CFavg) / P] * pdf * idf$$

Where: Ax = Area of peak
CFavg = Average Calibration Factor for that peak, from the curve
pdf = Prep Dilution Factor (Vf/Vi or Vf/Wi)
idf = Instrument Dilution Factor
P = number of peaks used

Concentration using Linear Regression (by external standard)

$$Cx = [\sum((Ax – b) / m) / P] * pdf * idf$$

Where: Ax = Area of peak
b = intercept, for that peak from the curve
m = slope, for that peak from the curve
P = number of peaks used
pdf = Prep Dilution Factor (Vf/Vi or Vf/Wi)
idf = Instrument Dilution Factor

CALIBRATION EQUATIONS

Calibration Factor is the ratio of the detector response (area) to the amount (mass or concentration) in the calibration standard.

$$CF = Ax / Cx$$

Where: Ax = Area of the compound
Cx = Concentration of the compound

Average CF or RRF = CFavg or RRFavg = $\sum(RFi) / n$

Where: RFi = Response or Calibration factor for each level
n = number of calibration points

Relative Standard Deviation (%RSD)
RSD is usually expressed in % and is a measure of the "goodness of fit" of a curve that uses the average response factor.

$$RSD = (SD / RFavg) * 100$$

Where: SD = Standard Deviation
RFavg = Average Calibration Factor or Relative Response Factor

$$\text{Standard Deviation} = SD = \sqrt{\{[\sum (RFi - RFavg)^2] / (n-1)\}}$$

Where: RFi = Response or Calibration factor for each level
RFavg = Average Calibration Factor or Relative Response Factor
n = number of calibration points

Linear Regression Equations
$y = mx + b$

Where: y = response (area of peak
x = concentration
m = slope
b = intercept

$$\text{Slope (m)} = [(\sum wx_iy_i * \sum w) - (\sum wx_i * \sum wy_i)] / [(\sum w * \sum wx_i^2) - (\sum wx_i * \sum wx_i)]$$

$$\text{Intercept (b)} = y_{avg} - (m * x_{avg})$$

Correlation Coefficient (r)

$$r = \frac{[(\sum w * \sum wx_iy_i) - (\sum wx_i * \sum wy_i)]}{\sqrt{\{[(\sum w * \sum wx_i^2) - (\sum wx * \sum wx_i)] * [(\sum w * \sum wy_i^2) - (\sum wy_i * \sum wy_i)]\}}}$$

$$\text{Coefficient of Determination } (r^2) = r * r$$

Where: x_i = individual values for the independent variable (concentration)
y_i = individual values for the dependent variable (response, area)
w = weighting factor (for no weighting w = 1)
x_{avg} = average of the x-values
y_{avg} = average of the x-values

Percent Difference (or Percent Drift), %D:
For calibration verification standards, the %D is the difference between the true concentration of the standard and the calculated concentration of the standard, divided by the true concentration, multiplied by 100:

%D (Percent Difference) = ((Cws - Cf) / Cws) * 100

Where: Cws = true concentration of the spiking standard
Cf = final measured concentration in the spiked sample

BATCH QC

Percent Recovery (%R):
The recovery is the measured concentration divided by the true concentration of the spike.

%Recovery = (Cf – Cs) / (Cws * Vws) *100

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard

Relative Percent Difference (RPD):
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

%RPD = |(Cs - Cdup)| / ((Cs + Cdup)/2) * 100

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

Make a Working Standard from a Source (Stock) Standard:
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where: Vss = Volume of Source Standard (mL) needed to make Working Standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

APPENDIX_2: SOURCE & WORKING STANDARDS

STANDARDS DOCUMENTATION

Source Standards
Source standards are those purchased directly from a chemical manufacturer or vendor. The LIMS S-name is unique to the manufacturer of the source standard; if a source standard is obtained from a different manufacturer, a new S-name must be assigned and the information entered in the "Standard Definitions" table before the standard can be assigned an S#.

Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is one year from date received. Certificates of Analysis should be obtained from the vendor of each source standard; the certificates should be labeled with the LIMS ID and the date received and filed in the 3-ring binder.

Enter all source standards into LIMS immediately upon receipt, using the Standards Menu "Standard Inventory", listing the date received, lot number, and expiration date. Write the LIMS S# on the vials and the certificate of analysis.

The standards listed below were those in use at the time this document was written, however standards may be purchased from different vendors so long as they are traceable through LIMS and the Standards Prep Logs.

Working Standards
Working standards are those prepared by C&T. For working standards, the LIMS S-name is not necessarily unique to the source standard used to create the working standard but *is* unique to the compound list and concentrations contained in the working standard; if the concentration or compounds in the working standard changes, a new S-name, compound list and concentrations must be entered in the "Standard Definitions" table before the standard can be logged in and assigned an S#. It is *very important* to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

Working standards expire 180 days after preparation from the source standards unless any of the source standards expire before the 180 days. If any of the source standards expire before the 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards. **The expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**.

In the Standards Benchbook, enter the prep date, LIMS S#, concentration, and volume of each source standard used, solvent name and lot#, the LIMS S-name and concentration of the working standard, expiration date, and prep chemist's initials.

In LIMS, use the "Standards Inventory" screen to enter the prep chemist's initials, prep date, and S# of the source or intermediate standards used to make the working standard; LIMS will then assign a standard number (S#).

SOURCE STANDARDS _____

Label each vial with the contents, LIMS S#, and expiration date. Store standards at 4°C (± 2°C) in Refrigerator #21 in the pesticides lab; standards may not be stored in a refrigerator containing samples or extracts.

Primary Source Standards

Analytes	Concentration (ug/mL)	Supplier & Catalog#	LIMS S-Name
Surrogates (TXCM/DCB)	200	Ultra Scientific ISM-320	PSS
Primer (TCL Pesticides)	2,000	Supelco 4-8913M	
Aroclor 1016/1260 Mix	1,000	Restek 32039	AR_16/60
Aroclor 1221	1,000	Restek 32007	AR21_R
Aroclor 1232	1,000	Restek 32008	AR32_R
Aroclor 1242	1,000	Restek 32009	AR42_R
Aroclor 1248	1,000	Restek 32010	AR48_R
Aroclor 1254	1,000	Restek 32011	AR54_R
Aroclor 1262	1,000	Restek 32409	AR62_R
Aroclor 1268	1,000	Restek 32410	Ar68_R

Second Source (ICV) Standards

Analytes	Concentration, in Hexane (ug/mL)	Supplier & Catalog#	LIMS S-Name
Aroclor 1016/1260 Mix	100	Ultra Scientific PPM-8082-1	1660_U
Aroclor 1221	100	Ultra Scientific PP-292-1	1221_U
Aroclor 1232	100	Ultra Scientific PP-302-1	1232_U
Aroclor 1242	100	Ultra Scientific PP-312-1	1242_U
Aroclor 1248	100	Ultra Scientific PP-342-1	1248_U
Aroclor 1254	100	Ultra Scientific PP-352-1	1254_U
Aroclor 1262	100	Ultra Scientific PP-372-1	1262_U
Aroclor 1268	100	Ultra Scientific PP-382-1	1268_U

WORKING STANDARDS PREPARATION

Because the solvent may dissipate over time, these solutions must be discarded after six months or sooner if there is a discrepancy when compared with other standards. Verify that the LIMS expiration date of the working standard does not exceed that of any of the source or intermediate standards used to make it. If any of the source standards expire before the 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards. **The expiration date of the working standard must not exceed the expiration date of any of the source standards from which it was made**.

Prepare the working standards in hexane, by diluting source standards to volume in a Class-A volumetric flask. Label the standards vials with the name & concentration (or calibration level) of the standard, LIMS S# and the expiration date. Store standards at 4°C (± 2°C) in the extraction lab refrigerator; do not store in a refrigerator containing samples or extracts.

Make up a 10,000 ug/L intermediate standard then dilute this standard in hexane to make the calibration standards.

Level	Add Vol. (mL) Std	Using Std Name	Using Std Conc (µg/L)	Final Vol. (mL) in Hexane	Final Conc (µg/L) of Aroclors / Surrogates	LIMS S-Name
ICAL Intermediate	1.00 1.00	AR_16/60 PSS	1,000,000 200,000	100	10,000 / 2,000	RES16/60_S
ICAL 1	0.100	RES16/60_S	10,000 / 2,000	100	10 / 2	PCB10_2
ICAL 2	0.250	RES16/60_S	10,000 / 2,000	100	25 / 5	PCB25_5
ICAL 3	1.00	RES16/60_S	10,000 / 2,000	100	100 / 20	PCB100_20
ICAL 4	2.50	RES16/60_S	10,000 / 2,000	100	250 / 50	PCB250_50
ICAL 5	5.00	RES16/60_S	10,000 / 2,000	100	500 / 100	PCB500_100
ICAL 6	7.50	RES16/60_S	10,000 / 2,000	100	750 / 150	PCB750_150
ICAL 7	10.0	RES16/60_S	10,000 / 2,000	100	1,000 / 200	PCB1K_200
ICV Intermediate	1.00 1.00	1660_U PSS	1,000,000 / 200,000	100	10,000 / 2,000	PCBULTRA_S
ICV	2.50	PCBULTRA_S	10,000 / 2,000	100	250 / 50	ULTRA_1660

Initial calibration curves for the remaining analytes are diluted in the same pattern as shown above and named in reference to the Aroclor (ie: Ar1232 would be 32_750, 32_500, etc). Aroclor 1232, Aroclor 1242, and Aroclor 1248 are analyzed as discrete standards. Aroclor 1221 and 1254 source standards may also be combined into a single standard (2154_750, 2154_500, etc).

The initial calibration verification (ICV) standard is prepared using a source standard obtained from a different manufacturer from the initial calibration curve standards. Continuing calibration

verification (CCV) standards are prepared from either the same manufacturer as the initial calibration curve or from a different manufacturer.

EPA 608 Method Modification: EPA 608 calls for the "stock standards" to be prepared in isooctane; C&T uses hexane because sample extracts end up in hexane and use of the same solvent improves matrix-matching between standards and samples.

Note: The USACE recommends that CCV's be either the same standards as the initial calibration standards or at least be prepared from standards obtained from the primary vendor, to more easily differentiate between instrument problems and variability of the standards.

Continuing Calibration Verification Standards (250µg/L Aroclor with 20µg/L TCMX/DCB): Each CCV standard is made by adding 2.50 mL of the primary source intermediate (containing 10,000 µg/L of Aroclor and 2000 µg/L of TCMX/DCB) to make a final volume of 100 mL in hexane. The final concentration of the CCV standard is 250 µg/L of the Aroclor and 50 µg/L TCMX/DCB. The primary source intermediates are named RES##_S and the CCV standards are named RES_##, where ## is the Aroclor number.

APPENDIX_3: PESTICIDE & PCB DILUTIONS

Let the extracts warm to room temperature then prepare the dilution in either an autosampler vial or an insert. See table below for appropriate volumes. Shake the dilution and invert 3 times to mix.

Dilution Factor	Made In	Extract Volume (µL)	Hexane Volume (µL)
2x	Insert	100	100
	GC vial	500	500
3x	Insert	50	100
	GC vial	250	500
4x	Insert	50	150
	GC vial	250	750
5x	Insert	40	160
	GC vial	200	800
10x	Insert	20	180
	GC vial	100	900
20x	Insert	10	190
	GC vial	50	950
50x	GC vial	20	980
100x	GC vial	10	990

SERIAL DILUTIONS

If you need to make a >100x dilution, first make the 100x dilution listed above, then make further dilutions, in hexane, using that as an intermediate.

Dilution Factor	Using Primary Dil'n	Made In	Extract Volume (µL)	Hexane Volume (µL)
200	100x	GC vial	100	100
500	100x	GC vial	40	160
1,000	100x	GC vial	20	180

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APPENDIX_4: INSTRUMENT CONDITIONS

The settings listed below were those in use at the time this document was written. Parameters may be changed at the analyst's discretion in order to optimize instrument performance. Changes are documented in the instrument's maintenance log.

GC-06 / GC-16	Hewlett Packard 5890	
Columns:	Front (Channel A):	Restek RTx-CLPesticides
	Back (Channel B):	Restek RTx-5
Column Insertion:	Injector side:	5 mm from the end of the ferrule
	Detector side:	70 mm from the bottom of the nut
Gases:	Carrier:	Hydrogen
	Makeup:	5% Methane in Argon (P5) at 50 mL/min

Oven

Oven On:	Yes
Equilibration Time(min):	0.2
Maximum Temperature (°C)	325

Ramp	Rate (mL/min)	Final Temp (°C)	Hold Time (min)
Initial	- -	140	0
1	7	270	0
2	25	325	3

Zone Temperature (°C)

Inj A:	205
Inj B:	205
Det A:	300
Det B:	300
Constant Flow (mL/min):	2.5

Front Injector

Sample Washes	3
Sample Pumps	2
Viscosity Delay	1
Solvent A washes	3
Solvent B washes	3

Valves

Valve 3:	On
Valve 4:	On

Inlet Purge Valves

Init State	On Time (min)	Off Time (min)
A	0.2	0
B	0	0

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GC-22 Varian CP-3800 w/ CP-8400 Autosampler

Columns:	Front (Channel A):	Restek RTx-CLPesticides
	Back (Channel B):	Restek RTx-5
Column Insertion:	Injector side:	3.7 cm from the bottom of the nut
	Detector side:	10.5 cm from the bottom of the nut
Gases:	Carrier:	Helium
	Makeup:	Nitrogen

Front Injector

Temperature (°C): 210

Split: Time (min)	State	Ratio
Initial	Off	0
0.5	On	100
2.0	On	50
2.5	On	5

Constant Flow Rate (mL/min):	2.2
Pressure Pulse Enable:	On
Pressure Pulse Pressure (psi):	30
Pressure Pulse Duration (min):	0.25

Middle Injector

Temperature (°C): 210

Split: Time (min)	State	Ratio
Initial	Off	0
1.2	On	100
2.7	On	50
25	On	5

Constant Flow Rate (mL/min):	2.2
Pressure Pulse Enable:	On
Pressure Pulse Pressure (psi):	30
Pressure Pulse Duration (min):	0.95

CP-8400 Autosampler

Injector Position:	1 & 2
Use Injector Delay (min):	0.7
Sample Penetration Depth %:	90
Solvent Penetration Depth %:	90
Number of Fill Strokes:	3
Fill Volume for Fill Strokes (µL):	5
Sample Flushes:	0
Pre-Inject Flushes:	3
Post-Inject Flushes:	1
Clean Solvent Source Vial:	1

Oven

Stabilization Time (min): 0.2

Step	Temp(°C)	Rate (mL/min)	Hold (min)
Initial	140	- -	0
1	280	7.5	0
2	310	25	4

Detector Settings:

Front:	Temperature (°C):	325
	Range:	1
	Range Auto-zero:	No
	Electronics:	On
	Time Constant:	Fast
	Cell Current:	N2 High
	Contact Potential:	300
Back:	Temperature (°C):	325
	Range:	1
	Range Auto-zero:	No
	Electronics:	On
	Time Constant:	Fast
	Cell Current:	N2 High
	Contact Potential:	300

This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

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GC-25 Varian CP-3800 w/ CTC Analytics Combipal Autosampler

Columns:	Front (Channel A):	Restek RTx-CLPesticides
	Back (Channel B):	Restek RTx-5
Column Insertion:	Injector side:	3.7 cm from the bottom of the nut
	Detector side:	10.5 cm from the bottom of the nut
Gases:	Carrier:	Helium
	Makeup:	Nitrogen

Front Injector

Temperature (°C): 200

Split: Time (min)	State	Ratio
Initial	On	10
0	Off	0
0.3	On	50
2.5	On	10
25	On	2

Constant Flow Rate (mL/min):	2.5
Pressure Pulse Enable:	On
Pressure Pulse Pressure (psi):	45
Pressure Pulse Duration (min):	0.23

Middle Injector

Temperature (°C): 210

Split: Time (min)	State	Ratio
Initial	On	10
0.94	Off	0
1.23	On	50
3.43	On	10
25	On	2

Constant Flow Rate (mL/min):	2.2
Pressure Pulse Enable:	On
Pressure Pulse Pressure (psi):	30
Pressure Pulse Duration (min):	0.95

Oven

Stabilization Time (min): 0.2

Step	Temp (°C)	Rate (mL/Min)	Hold (min)
Initial	110	- -	0
7	280	1.72	26.01

Detector Settings:

Front:	Temperature (°C):	325
	Range:	10
	Range Auto-zero:	No
	Electronics:	On
	Time Constant:	Fast
	Cell Current:	N2 High
	Contact Potential:	-400
Back:	Temperature (°C):	325
	Range:	1
	Range Auto-zero:	No
	Electronics:	On
	Time Constant:	Fast
	Cell Current:	N2 High
	Contact Potential:	330

CombiPal Autosampler Settings

Cycle:	GC-DUAL
Syringe:	10 µL

1.Sample Vol:	1.0 µL	Pre Cln Slv1:	1	Fill Volume:	5.0 µL
1.Air Vol:	0 nL	Pre Cln Slv2:	0	Fill Speed:	2 µL/s
1.Inject to:	FRONT	Pre Cln Spl:	1	Fill Strokes:	3
1. Inj Time Diff:	0s	Int Cln Slv1:	0	Pull up Del:	1s
2.Sample Offs:	0	Int Cln Slv2:	0	Inject Speed:	100 µL/s
2.Sample Vol:	1.0 µL	Pst Cln Slv1:	0	Prep Inj Del:	200 ms
2. Air Vol:	0 nL	Pst Cln Slv2:	3	Pst Inj Del:	200 ms
2. Inject to:	MIDDLE				

This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

APPENDIX_5: MAINTENANCE & TROUBLE-SHOOTING

Any maintenance performed on the instrument must be documented in the Maintenance logbook. This notation must include the reason the maintenance was necessary ('CCV failing', etc.), the date, analyst initials, maintenance steps performed, and resolution ('ICAL passed', etc.) of the maintenance.

"SAFETY SHUT-DOWN":
A "safety shut-down" will occur when the EPC (electronic pressure control) can not reach the pressure set-points due to problems with the gas supply. The problem may be due to a cored septum, a faulty valve, someone disconnecting a tank incorrectly, a loose injection port screw, a broken or plugged column, or an empty tank. Investigate and correct the problem before restarting the sequence.

The GC will begin beeping up to 2 minutes before the "safety shut-down" happens. If the problem is immediately corrected, the shut-down may be averted. If this happens, watch the sequence to ensure that it does continue correctly.

To restart GC-06 after a safety shut-down, turn off the GC, wait about 15 seconds, then turn the GC back on.

TROUBLE-SHOOTING

Symptom: **Dirty instrument blank**

Possible causes:

- Semivolatile or nonvolatile sample residues in injector. Remove injector liner and stainless steel seal and swab out injection port with a Q-Tip soaked in Hexane. Change septum, injector liner and gold seal. Reanalyze instrument blank.

- Semivolatile or nonvolatile sample residues in column. Remove guard column from injector and cut off about 1 loop. Reinstall. Reanalyze instrument blank. If instrument blank is still dirty, rinse column and reanalyze instrument blank. See RESTEK literature for column rinsing instructions.

PREVENTATIVE MAINTENANCE _____

1.) Septa should be replaced after no more than 100 injections.

2.) Injector liners should be replaced whenever a CCV is out of limits or you cannot get a clean instrument blank.

3.) Check the tightness of the capillary column detector adapter nut weekly. Replace the ferrule in the adapter when you can no longer tighten the fitting and the adapter is still loose.

4.) Check tightness of capillary column ferrule nuts in detector and inlet fittings; they should be snug - do not over-tighten.

5.) Autosampler syringes should be removed from the autosampler and cleaned weekly. Clean by drawing and expelling a 1:1 vol:vol Hexane/Acetone mixture. After 3 rinses, withdraw the plunger and wipe it with a Kimwipe. Repeat; continue until no more residue is being wiped from the plunger.

6.) Detectors may need thermal cleaning (bake out) after running many "dirty" samples. They should be baked out about every three months whether they need it or not. Warning - baking out a detector may change its response!

To thermally clean an EC detector:

10.5 Remove the column from the detector and cap the detector.

B.) Set DET A TEMP and DET B TEMP to 375°C

C.) Monitor the progress of the cleaning by observing the detector signals (SIG 1 or SIG 2). Typically the signal will rise as the detector temperature goes to 350°C. If the detector is dirty the signal will peak and decrease to a constant level. If the detector is not too dirty the signal may just rise until the detector temperature equlibrates and then stay constant. Thermal cleaning is complete when the detector signal has been constant for about 15 minutes.

D.) Reset DET A TEMP and DET B TEMP to 325°C

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APPENDIX_6: RETENTION TIMES & ELUTION ORDER

Retention Time Windows: "Retention Time Windows" are necessary because compounds may not elute at *exactly* the same time during each and every injection, due to slight variations in temperature, flow rate, or injection composition (sample viscosity, compound concentrations), etc. The RT-window is the length of time (width, in minutes, on the chromatogram) during which any peak eluting within the window is presumed to be the analyte of interest. "72-hour RTW Study" is a term often used by auditors to describe statistical analysis of the retention times of standards injected over a 72 hour sequence; theoretically, the RT windows determined by this study can be used for routine analysis, however the studies that C&T has conducted in the past result in windows that are too narrow for routine use. C&T therefore uses the default retention time windows of ± 0.03 minutes as specified in EPA 8000B.

If a 72-hour RT study is required by a client or auditor, the RT windows are defined as plus or minus three times the standard deviation of the absolute retention times for each compound in the calibration standard mix as measured over the course of 72 hours. (*Note:* This procedure has historically (and consistently) produced RT-windows too tight for routine use.) In the event that a standard deviation is 0.00, then use the 0.03-minute window (see 8000B). However the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

For multi-component standards, i.e. PCB's, the analyst should use the retention time windows but also rely on pattern recognition.

Absolute Retention Times: The "absolute" retention time of any compound is the expected time of the compound is the center of the RT window. Use the retention time for each analyte from the mid-level initial calibration standard as the "absolute" retention time. Examine the daily CCV retention times and verify that the peak maximum falls at the center of each retention window. If any of the compounds fall outside their daily or approach the limit of the window, update the retention times based on the first CCV of that sequence. Save the method file with the new date and process the entire sequence with the new RT's.

Method Modification Note: EPA 8000B, Section 7.6.5 suggests updating the absolute retention times each time a new sequence is started. Because the retention times for these compounds are relatively stable, C&T has only found it necessary to update the retention times when performing the initial calibration or when an RT-shift is observed in the CCV.s.

Elution Order: The order in which compounds elute is based on chemical composition of the stationary phase of the column and on the instrument conditions (flow rates, temperature programming, column length). Given a specific set of instrument conditions (flow rates, temperature program) the order in which compounds elute from a column should remain constant but may differ between different types of columns.

APPENDIX_7: INTERFERENCES & MULTICOMPONENT CHROMATOGRAMS

Matrix interferences may cause inaccuracies in the determined element concentrations. These interferences may interfere with the integration of the data or with the actual instrumental analysis of the samples.

Sulfur

The presence of elemental sulfur is generally indicated by the presence of several large peaks that may obscure the target analytes or otherwise interfere with the detection and integration of the target analytes; see the following pages for example chromatograms. This is a common interference for most soil and wastewater samples but is not as prevalent in groundwater samples. If sulfur is present, it should be removed using copper powder and the extracts should then be reanalyzed.

Multicomponent Pesticides & PCB's

PCB's, technical chlordane, and toxaphene are all multi-component (ie: consist of more than a single compound) analytes that will be observable on the sample chromatograms and may interfere with quantitation of discreet PCB congeners or pesticides; see the following pages for example chromatograms. If these multi-components are present, it may not be possible to reliably report a single, undiluted run; see the Department Manager or QC Chemist for guidance.

Organic Acids

Organic acids do not appear on the sample chromatograms but are indicated by the disappearance of DDT, Methoxychlor, *and their breakdown products* from pesticide CCV's. These interferences are common to wastewater treatment facilities and municipal landfills. Any sample extracts suspected, or known through site history, to contain organic acids should be run at a 100x or more dilution. If these extracts are analyzed at a less dilute level, the acids will destroy the column's stationary phase and the entire column will then need to be replaced.

Hydrocarbons

Hydrocarbons present in dark or oily extracts will cause active sites in the injection port, leading to breakdown problems. See the "Dilutions" section in the main body of this SOP for further discussion.

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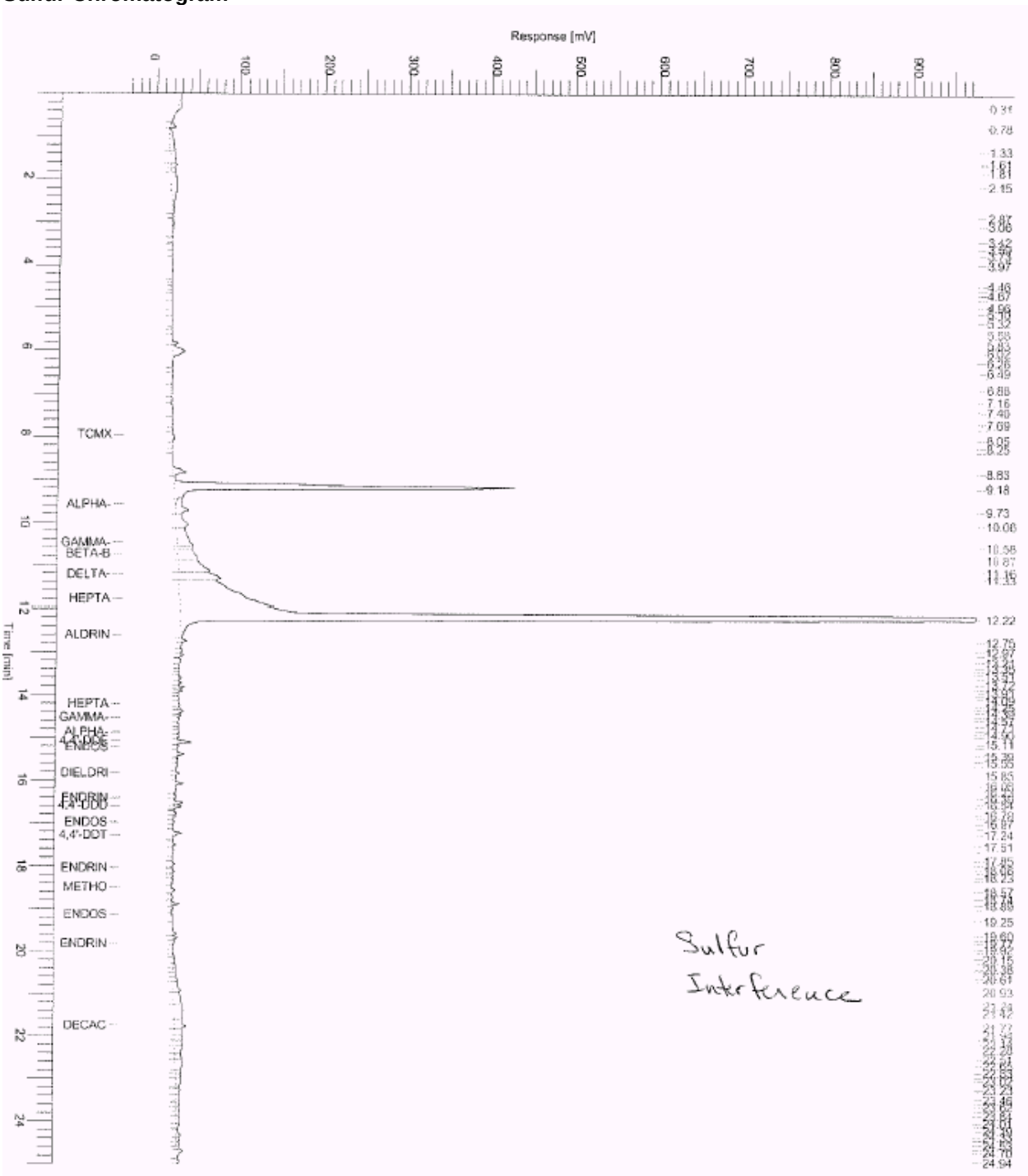
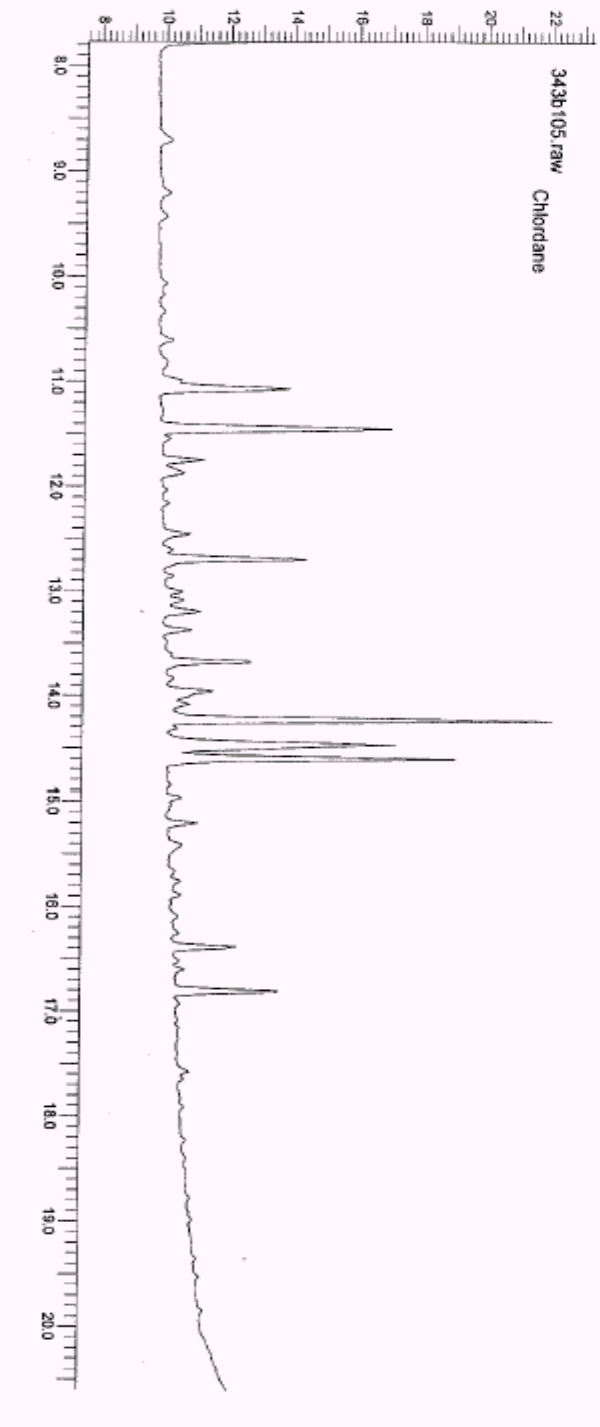
Sulfur Chromatogram

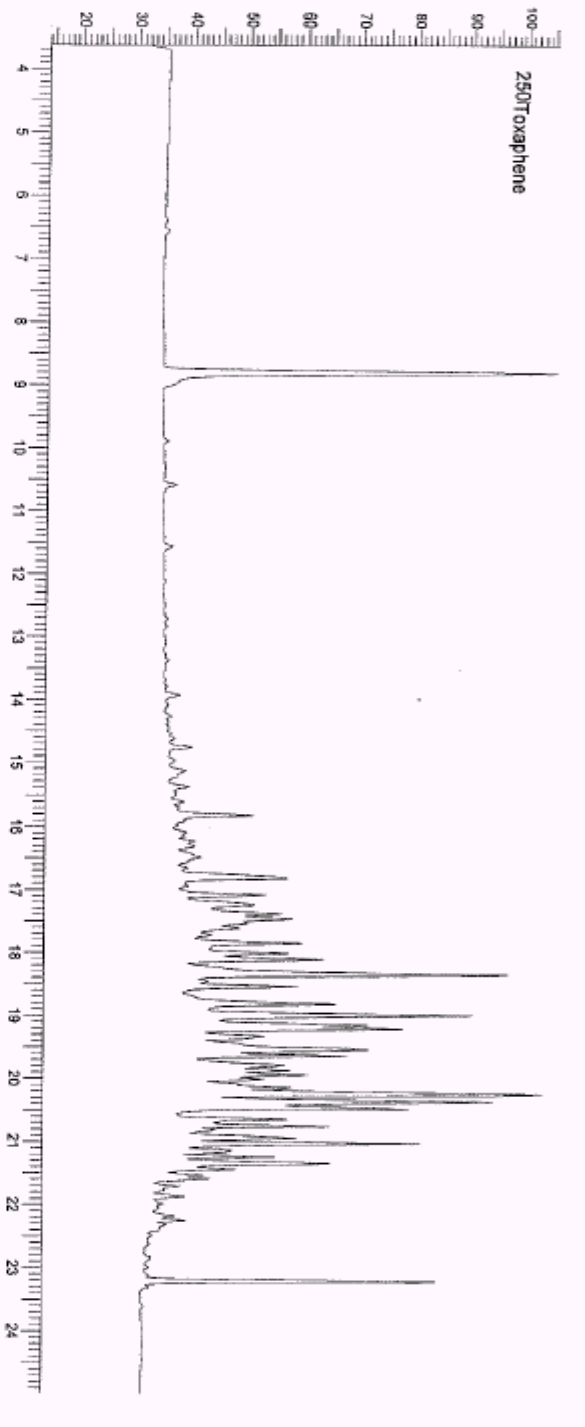


Curtis & Tompkins, Ltd

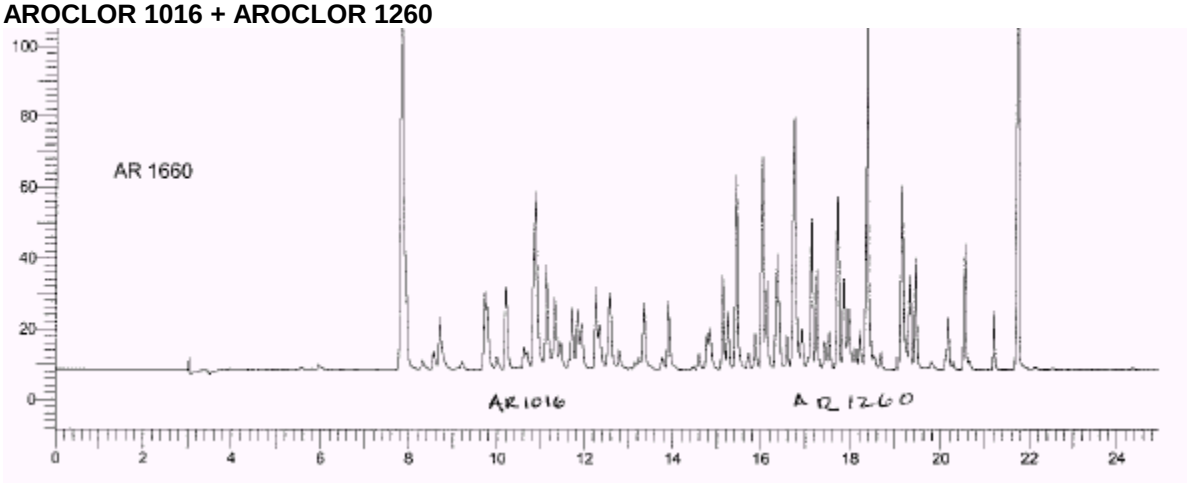
Technical Chlordane Chromatogram



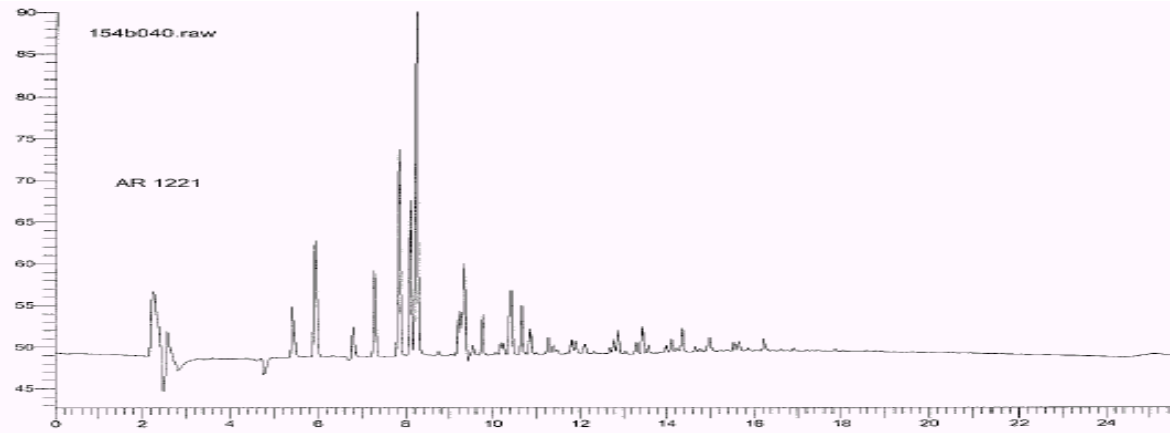
Toxaphene Chromatogram



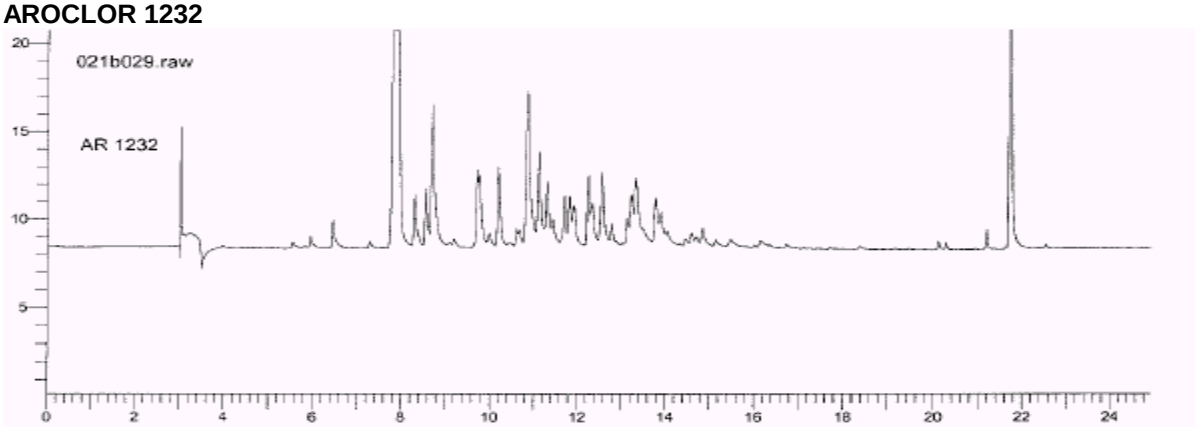
AROCLOR 1016 + AROCLOR 1260



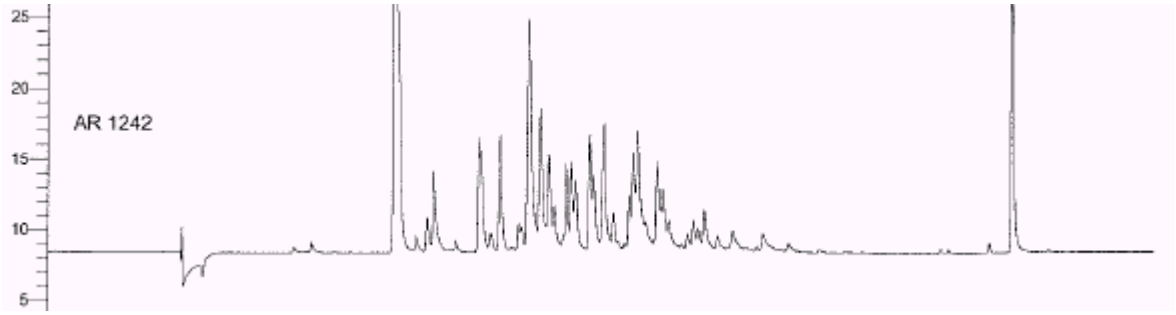
AROCLOR 1221



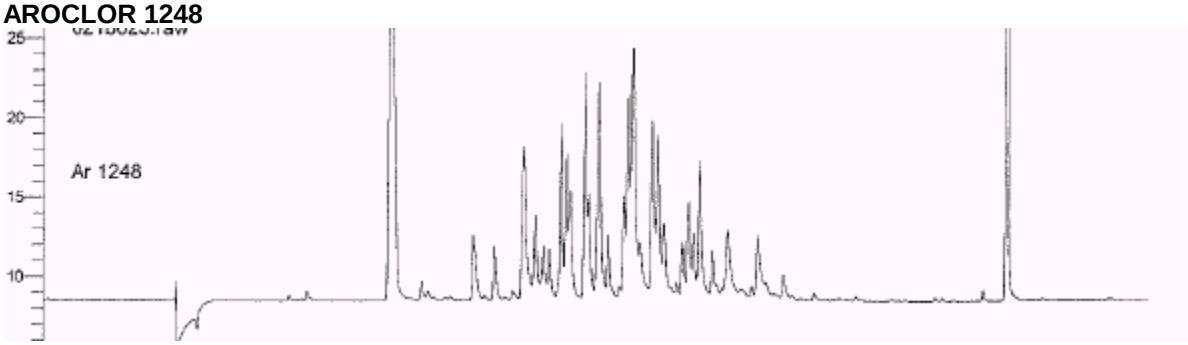
AROCLOR 1232



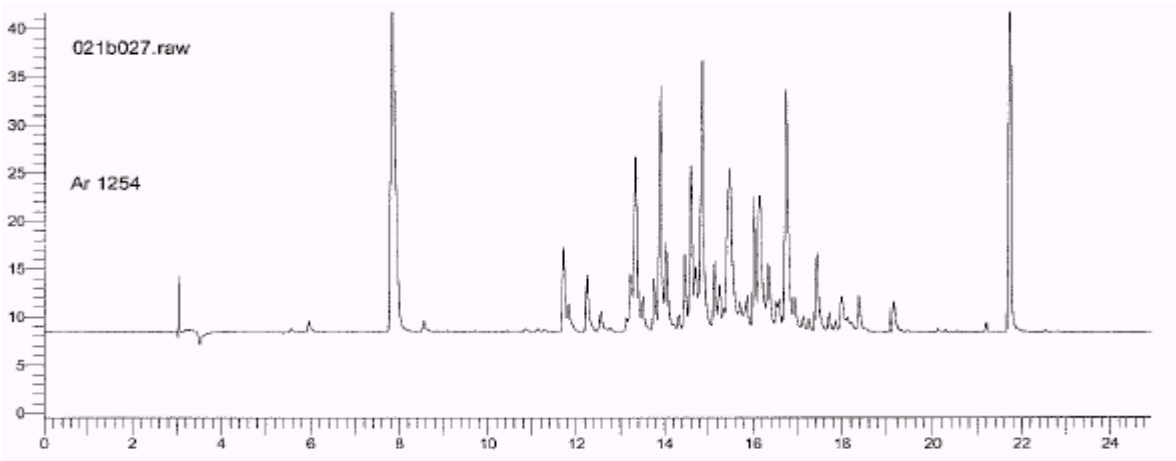
AROCLOR 1242



AROCLOR 1248



AROCLOR 1254



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APPENDIX_8: TARGET COMPOUNDS & REPORTING LIMITS

CAS #	Compound	Water RL µg/L	Soil RL µg/Kg	Wipe RL µg/wipe
12674-11-2	Aroclor-1016	0.5	12	2.5
11104-28-2	Aroclor-1221	1	24	5.0
11141-16-5	Aroclor-1232	0.5	12	2.5
53469-21-9	Aroclor-1242	0.5	12	2.5
12672-29-6	Aroclor-1248	0.5	12	2.5
11097-69-1	Aroclor-1254	0.5	12	2.5
11096-82-5	Aroclor-1260	0.5	12	2.5

Additional Compounds (may be added to the compound list):

37324-23-5	Aroclor-1262	0.5	12	2.5
11100-14-4	Aroclor-1268	0.5	12	2.5

Surrogates:

2051-24-3	Decachlorobiphenyl (DCB)
877-35-2	Tetrachloro-m-xylene (TCMX)

Reporting Limits may vary, depending on the volume submitted for analysis, preparation method used, and %moisture (if reported on a dry-weight basis). The reporting limits listed above are based on the following prep volumes & weights:

Water Samples:	Initial Volume = 1,000 mL	Final Extract Volume = 25 mL
3550 Soil Samples:	Initial Weight = 30 g	Final Extract Volume = 25 mL

This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

APPENDIX_9: **INITIAL CALIBRATION**

Procedure & Acceptance Criteria

REQUIREMENTS

An initial calibration curve must be analyzed whenever instrument conditions (temperature programs, flow rates, etc.) have been changed, the detector has been cleaned, or when a new column has been installed. In general, a new calibration curve must be made whenever instrument conditions have been altered, or whenever the continuing calibration verification no longer passes acceptance criteria.

The instrument analytical range must be established by running a minimum of 5 calibration standards, containing Aroclor 1016 and Aroclor 1260, at levels that bracket the quantitation range (see Appendix_2 for standard suppliers); the lowest standard must be at or below the reporting limit and the highest standard determines the upper end of the quantitation range. The standards must be analyzed in order of increasing concentration. Points may be dropped so long as the following criteria are met:

- The highest concentration standard may be omitted so as long as there are at least five points remaining and the remaining highest point defines the top of the calibration range (any extracts which exceed this response must be diluted and reanalyzed).

- If the high point of a single congener in the Aroclor needs to be dropped, the high point should be dropped for each of the other peaks as well to consistently define the top of the linear range for the entire Aroclor.

- The lowest concentration standard may be omitted from curve if, *and only if*, the resulting lowest standard is at or below the reporting limit for samples and there are at least five points remaining. For surrogates, the lowest concentration standard must be $\leq$10x of the level used to spike the samples and QC.

- Mid-point standards may not be omitted simply to improve the RSD or linear correlation coefficient. They may, however, be reanalyzed if a poor injection is suspected. The reanalysis must occur immediately after the curve so long as no sample extracts were analyzed since the last calibration standard and all compounds are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular compound.

The curve must be verified by analyzing an Initial Calibration Verification (ICV) standard comprised of standards obtained from a different manufacturer than those used to prepare the ICAL standards. See Appendix_1 for calculation of response factors, RSD, and correlation coefficient.

Initial Calibration Levels (pg)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	10	25	100	250	500	750	1,000
Aroclor 1260	10	25	100	250	500	750	1,000
Decachlorobiphenyl (DCB)	2	5	20	50	100	150	200
Tetra-chloro-m-Xylene (TCMX)	2	5	20	50	100	150	200

Note: EPA 8082 Section 5.6.1 specifically allows the use of an Ar1016/1260 mix to establish the linearity of the detector and a single-point initial calibration to establish the response factor for the remaining Aroclors, however EPA 608 and many Department of Defense (USACE, Navy, AFCEE) projects require a multi-point calibration if the pattern is detected in any of their samples. Check the project notes to verify the requirement for samples associated with any DoD project.

PROCEDURE

For each of the Aroclors, select three to five representative peaks as the basis of quantitation and assign a unique identification (i.e.'PCB1260 #1', 'PCB1260 #2', etc.) to each peak. LIMS will calculate the calibration factor for each of these peaks in each of the calibration standards.

If the retention *times* have changed significantly, analyze the 5-point Ar1016/1260 along with a single-point for each of the other Aroclors, for pattern recognition. If samples are being analyzed by EPA 8082 and one of the other Aroclors is identified in the extracts, the calibration verification standard immediately preceding the sample can be used to used to establish the calibration factor for that Aroclor, so long as the 1016/1260 ICAL has demonstrated the linearity of the detector (as per 8082 Section 5.6.2).

EPA 608 *Method Note:* If samples are being analyzed for EPA 608, a multi-point calibration must be run for any of the other analytes if they are detected in the sample.

Run the Initial Calibration Standards:

1.) Prepare the standards as described in Appendix 2.

2.) Prepare an Initial Calibration Verification (ICV) standard from source standards obtained from a different manufacturer than the ICAL standards.

3.) Perform any needed instrument maintenance and run a "primer" at about 2,000 ug/L followed by a hexane instrument blank. If any target compound is detected above the reporting limit, run another instrument blank.

4.) Load the calibration standards onto the autosampler tray in order of increasing concentration.

5.) Add instrument blanks before and after the ICAL standards to demonstrate that the low-level standard was not influenced by instrument contamination and that analytes at the high-level concentrations will not carryover into real-world samples.

6.) If the retention times of the analytes in the new calibration curve are expected to be significantly different than the previous calibration curve, due to instrument maintenance such as column maintenance or changed flow or temperature parameters, also include a single calibration standard of each of the other Aroclors in the sequence.

7.) Load the ICV after the instrument blank that follows the calibration standards. The ICV, prepared from standards obtained from a second manufacturer, must be analyzed to verify that the standards used to create the initial calibration curve were prepared correctly.

8.) In the sequence spreadsheet, make sure the new method is entered. To change the method, select the first box in the METHOD column and click the green diamond button. Slect the new method and click OPEN. Right click on the first box in the METHOD column and select FILL DOWN.

Note: Always make sure the new method is created and entered into the sequence BEFORE processing an ICAL or updating retention times. This ensures that you do not change and overwrite the old method.

9.) In the SAMPLE ID column, enter ICAL points below, identifying the type of sample as initial calibration standards, the LIMS identification of the standards, and the applicable dilution factors. The “stype” and S# must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information.

primer
hexane
ICAL, S#, PCB10_2
ICAL, S#, PCB25_5
ICAL, S#, PCB100_20
ICAL, S#, PCB250_50
ICAL, S#, PCB500_100
ICAL, S#, PCB750_150
ICAL, S#, PCB1000_200
hexane
ICV, S#, ULTRA_1660

Where:
S# is the LIMS S# of the standard used

10.) Save the sequence by going to FILE > SEQUENCE > SAVE.

11.) Run the ICAL, following the steps on "Running a Sequence in EZChrom" as described in Appendix_11.

Process a Multi-Point ICAL in EZChrom:

12.) Make sure that you have created and entered a new method into the sequence before processing an ICAL. Also make sure the new method is currently opin in your EZChrom session.

13.) Check the integrations of the Initial Calibration data files and work up the data using the integration events outlined in Appendix_11. Make sure all of the analytes are correctly identified for every calibration level on both channels. If you need to change the retention times, see "Updating the Retention Times" in Appendix_11.

Peaks should be integrated from baseline to baseline. Manual integrations of any kind must be substantiated and documented on the Initial Calibration Report. Manual integrations must be consistently applied to all ICAL, CCV, and sample integrations. **Unsubstantiated alteration of peak integration solely to pass calibration criteria is illegal and is grounds for immediate termination.** If the reason for the integration is not intuitive and obvious, the analyst must document the reason on the data.

14.) Once the chromatograms are properly integrated, process the data. In the sequence spreadsheet, change the run type by selecting the row for the run and clicking the blue triangular button under the RUN TYPE column. The Sample Run Type(s) window should open. Check CLEAR CALIBRATION AT LEVEL and click OK. Change all of the ICAL runs to this run type.

15.) In the LEVEL column, enter the appropriate calibration level.

Note: To find the calibration level, open the "Peaks & Groups" table by going to METHOD > PEAKS/GROUPS. There is a column for each level with the spike amount entered for the relevant analytes.

16.) Process the ICAL by going to SEQUENCE > PROCESS. Type the run numbers for the ICAL runs in RANGE and make sure the box for PRINT METHOD REPORTS is checked.

Caution: Processing the ICAL will overwrite the old calibration in the method. Make sure that the new method is entered into the sequence before processing!

DATA REVIEW & APPROVAL

Review the calibration in EZChrom before processing it in LIMS.

17.) Go to METHOD > REVIEW CALIBRATION to verify that the calibration is linear and that the upper levels are not saturated. Click the name of the analyte to see the %RSD for that compound. To switch channels, use the drop down list on the main toolbar.

18.) Verify that the calibration is useable by reviewing the results against the following criteria:

18.1) The Relative Standard Deviation (RSD) of the Response Factors for both columns should be less than or equal to 20%.

EPA 608 *Method Note:* For EPA 608, the RSD must be ≤10%

18.2) If the RSD fails for any compound, compute the "grand mean" of the RSDs for all target compounds; if this mean RSD is less than or equal to 20% then the average response calibrations may be validated (see EPA 8000B Section 7.5.1.2 for details).

Note: For Department of Defense (Army Corp, Navy, AFCEE) projects, no individual compound's RSD may be > 30% (DoD QSM, Box 33 and Table B-2).

EPA 608 *Method Note:* For EPA 608, the RSD must be ≤10% and the method does not allow use of this "Grand Mean" concept; each analyte must meet the 10%RSD criteria.

18.3) If the average response factors fail to meet the 20% RSD or mean RSD criteria, employ a linear regression or quadratic model. If linear regression is used, a minimum of five points is required with a correlation coefficient $r \geq 0.995$ If a quadratic fit is used, a minimum of six points is required with a coefficient of determination $r^2 \geq 0.990$.

18.4) For each compound:

- The low point may only be rejected for those compounds that have reporting limits greater than that level.
- The high point may be rejected for compounds that tend to saturate at high levels so long as there are at least 5 points remaining for each compound in the ICAL.
- If a single point in the curve is causing the failure, the standard may be reanalyzed, so long as it immediately follows the original curve and all compounds are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular compound.
- The %D for recalculated concentrations should be within 20% of the true concentration of the standard (8000C requirement).

19.) Save the newly calibrated method by going to FILE > METHOD > SAVE.

20.) Open the Initial Calibration Verification (ICV) data file and verify that all compounds are integrated correctly. Process the ICV with the newly calibrated method.

The ICV verifies that the calibration standards were prepared correctly and highlights any discrepancies between the primary- and second-source standards.

The ICV should meet the CCV criteria of ≤ 15%D. If the %D for any analyte is outside this limit, calculate the mean %D for all compounds. If this is less than 15% then the CCV is considered acceptable.

Note: For Department of Defense (Army Corp, Navy, AFCEE) projects, no individual compound's %D may be > 20% (DoD QSM, Box 33 and Table B-2).

If the first ICV does not meet the acceptance criteria, another ICV standard may be analyzed; "x" out the first ICV and process the data from the second ICV. *Do not* "cherry pick" some compounds from the first ICV and others from the second ICV; if the second ICV is processed and used, all compounds must be taken from the second standard.

Processing an ICAL in LIMS:
After you've verified that the calibration passes all acceptance criteria, process the ICAL in LIMS.

21.) Open a web browser and go to the main Pesticides/PCB page.

22.) Under "Recent Sequences", choose the GC and then choose the sequence you want to process.

23.) Check the boxes next to each of your ICAL data files. Click the CREATE CALIBRATION button at the bottom of the screen.

24.) When the Calibration Editor opens, unselect any analytes that are not being calibrated (ex: Toxaphene and Technical Chlordane).

25.) Type a name for the calibration (ex: gc21pest_051, where 051 was the sequence on gc21).

26.) Click SAVE + PUT INTO USE and return to the sequence.

27.) At the top of the sequence, the newly created calibration should be listed. Click REVIEW to open the Review App for the calibration.

28.) Sign and review each of the runs. See Appendix_12 for guidelines on using the LIMS Review App.

Packaging the ICAL for Review:
29.) Print the LIMS ICAL summary. Click the calibration name at the top of the sequence. Go to the bottom of the on-screen ICAL summary and click the PRINT button.
30.) Print the EZChrom Calibration Report by going to METHOD > REVIEW CALIBRATON.

31.) Right click in the box at the top left with the list of analytes and select PRINT ALL PEAKS/GROUPS to print a Calibration Report for each analyte.

32.) Switch to the other EZChrom channel and print the Calibration Report for that channel.

33.) Verify that the LIMS and EZChrom calibration factors match.

Note: Any corrections to the ICAL must be done through EZChrom and then printed to ezchrom_capture to send the new data to LIMS. A new ICAL must then be created in LIMS. Any data processed with the incorrect ICAL would then need to be reprocessed against the corrected ICAL.

34.) Complete "GC & HPLC ICAL Review Checklist" and attach the EZChrom and LIMS calibration summaries. Turn the data in to the Group Leader, Department Manager or QC Chemist for review; the ICAL cannot be used to process final forms through LIMS until it has been reviewed and approved in LIMS.

Note: The initial calibration must be reviewed and 2nd-party approved in LIMS before final sample Form 1's can be printed; any forms printed before the ICAL is marked 'reviewed' will be flagged as draft results.

APPENDIX _10: CONTINUING CALIBRATION VERIFICATION (CCV)

REQUIREMENTS

Continuing Calibration Verification (CCV) standards are analyzed to verify that the response of the instrument has not changed significantly and that the curve may still be used to quantitate sample results. A CCV must be run at the beginning of each sequence before any samples are analyzed, after every 10 samples (or within 12 hours, whichever comes first), and at the end of the analytical sequence. The concentration of the CCV must be varied within the calibration range but should not be analyzed at either extreme (highest or lowest point) of the ICAL curve.

LIMs identifies samples that are associated with the DoD QSM requirements, other DoD requirements or commercial clients. It identifies all project specific criteria that are reviewed by the analyst before sample analysis.

For sequences containing samples that are not associated with requirements from DoD QSM, if the first CCV does not meet the acceptance criteria, another CCV standard should be analyzed. If two CCV's were analyzed, examine the first one against the acceptance criteria; if it fails, "x" out the first CCV, change the second to stype "CCV" and process the data from the second CCV. *Do not* "cherry pick" some compounds from the first CCV and others from the second CCV; if the second CCV is processed and used, all compounds must be taken from the second standard.

If the second analysis of the standard also fails to meet these criteria and the analyst suspects that the CCV standard has degraded, a different CCV standard may be analyzed once. If this standard passes, discard the standard that has been degraded. If the different CCV standard also fails, instrument maintenance required and recalibration may be required if major instrument maintenance is performed.

For sequences where both DoD QSM samples and non-DoD QSM samples are analyzed, both sets of CCVs will be run and integrated and both CCVs must pass the DoD QSM criteria. If the entire sequence contains DoD QSM samples only, then only one CCV will be analyzed.

See Appendix 9 for the Initial Calibration procedure and acceptance criteria. See Appendix 1 for calculation of %D and the calibration factor (CF).

ACCEPTANCE CRITERIA

Process the CCV through LIMS then examine the summary form against the following criteria to determine whether the CCV is acceptable:

1.) All compounds must fall within its retention time window and be automatically identified on both columns by the data system.

2.) All compound responses should be within 15% of the initial calibration (%D $\leq$ 15%).

3.) If the %D for an individual compound fails acceptance criteria, data may be reportable based on the following criteria:

a.) If the failing compound is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b.) If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c.) If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples should be reanalyzed.

d.) If the compound fails the %D criterion due to a low response and *was* detected (even below the reporting limit), the sample should be reanalyzed.

If any of the above criteria are not met, examine the integration to verify that each peak was correctly integrated. Manual integrations must be consistently applied to ICAL, CCV, and sample integrations. If manual integrations are performed, the file should be resent to LIMS so that a new CCV summary form can be generated.

WARNING: **Unsubstantiated alteration of peak integration solely to pass calibration or QC criteria is <u>illegal</u> and is grounds for immediate termination.**

APPENDIX_11: USING EZCHROM

Setting up a Sequence on EZChrom:

1. Open EZChrom Elite.

   1.1 Click PCB/PEST LAB.

   1.2 Right click on the GC that will run and select either OPEN (online) or OPEN OFFLINE.

   1.3 In USER NAME and PASSWORD, type the appropriate information.

   1.4 In PROJECT, find the GC you are for which you are opening the session.

   1.5 Click LOGIN.

   Note: You can write a sequence in either an online or offline session, but you can only run the sequence in an online session.

2. Open the latest sequence.

   2.1 Go to FILE > SEQUENCE > OPEN.

   2.2 Choose the latest sequence filename (###.seq).

   2.3 Click OPEN.

   This sequence will be used as the template to set up the next sequence.

3. Create a copy of that sequence.

   3.1 Go to FILE > SEQUENCE > SAVE AS.

   3.2 In “File Name”, type in the current Julian date.

   3.3 Click SAVE.

4. Change the filenames to use the current Julian date.

   4.1 Right click on the first box in the FILENAME column.

   4.2 Select FILL DOWN.

   4.3 In the DATA FILE box, type the Julian date, underscore, and the code for LINE NUMBER (eg 167_<###>).

   4.4 Click OK.

5. Re-number the vials under the VIAL column by right-clicking the first box and selecting FILL DOWN.

   5.1 Check INCREMENT VIALS, enter the vial placement of the starting vial, and increment by 1.

6. In the VOLUME column, enter the appropriate injection volume in µL (see Appendix_4 for the injection volume) and FILL DOWN.

   Note: The CombiPal Autosamplers are not controlled by EZChrom so the sequences run with these autosamplers do not use the VIAL and VOLUME columns. These parameters must be entered in the method on the autosampler itself.

7. In the sequence spreadsheet, make sure that that current method is being used.

   7.1 To change the method, select the first box in the METHOD column and click the green diamond button.

   7.2 Select the appropriate method (e.g. pest-run-###.met) and click OPEN.

   7.3 Right click on the first box in the METHOD column and select FILL DOWN.

8. In the SAMPLE ID column, enter what will run in the sequence in the format seen in the following example of a sequence table:

Run Type	Level	Vial	Volume (µL)	Sample ID	Method	Filename
Unknown	0	1	0.5	Primer	pcb-run-<###>	<###>-001
Unknown	0	2	0.5	Hex	pcb-run-<###>	<###>-002
Unknown	0	3	0.5	CCV,S<#>,pcb250_50	pcb-run-<###>	<###>-003
Unknown	0	4	0.5	CCV,<S#>,ar2154	pcb-run-<###>	<###>-004
Unknown	0	5	0.5	Hex	pcb-run-<###>	<###>-005
Unknown	0	6	0.5	MB,QC<###>,<batch#>	pcb-run-<###>	<###>-006
Unknown	0	7	0.5	LCS,QC<###>,<batch#>	pcb-run-<###>	<###>-007
Unknown	0	8	0.5	<sample#>,<batch#>	pcb-run-<###>	<###>-008
Unknown	0	9	0.5	MS,QC<###>,<batch#>	pcb-run-<###>	<###>-009
Unknown	0	9	0.5	MSD,QC<###>,<batch#>	pcb-run-<###>	<###>-010

   "Run Type" and "Level" should be "Unknown" and "0" respectively, unless an initial calibration curve is being run; see Appendix_9 for instructions on processing calibrations using EZChrom.

   The "Vial" and "Volume" columns are not used for sequences run on a CombiPal Autosampler.

20. Save the sequence by going to the menu and clicking FILE > SEQUENCE > SAVE.

Running a Sequence on EZChrom:

1. Open an online session on EZChrom for GC you want to run.

2. Open the sequence you want to run.

   2.1 Go to FILE > SEQUENCE > OPEN.

   2.2 Choose the sequence filename (###.seq).

   2.3 Click OPEN.

3. Start the sequence.

   3.1 Going to CONTROL > SEQUENCE RUN.

   3.2 In the RANGE box, type the range of the sequence you want to run.

3.3 Click START (or SUBMIT if the GC is already running and acquiring data).

4. You will need to start the CombiPal Autosampler (GC-21 & GC-25) manually because EZChrom does not control that feature.

Setting up the Sequence on the CombiPal Autosampler:

1. The main screen on the CombiPal controller should list the Job Queue. There should already be a job listed from the previous run. Turn the silver knob to outline the job. Press the silver button to select the job for editing.
2. Change the position of the first and last vials to be run. Turn the knob to outline FIRST and press the silver button to select. Turn the knob until the number reaches the first vial number you want to run. Repeat the process with LAST to set the last vial you want to run.
3. Press F4 to select HOME and return to the Job Queue menu.
4. Press F4 to select START to start the autosampler; there may be a slight delay.

Note: Remember to start the Sequence Run on EZChrom before starting the autosampler, otherwise the autosampler may inject and start the GC run before EZChrom is ready to acquire the data.

Processing Data on EZChrom:

1. Open EZChrom an OFFLINE session for the GC from which you will process data.

   Note: You can process data in an online session, however, it is best to have only one online session open for each GC at a time to ensure that only one user is running a sequence on that GC.

2. Open the sequence you want to run.

   2.1 Go to FILE > SEQUENCE > OPEN.

   2.2 Choose the sequence filename (###.seq).

   2.3 Click OPEN.

3. EZChrom sessions usually opens with a default “untitled” method, so you will need to select the correct method.

   3.1 Open the current method by going to FILE > METHOD > OPEN.

   3.2 Choose the method filename (pset-run-###.met).

   3.3 Click OPEN.

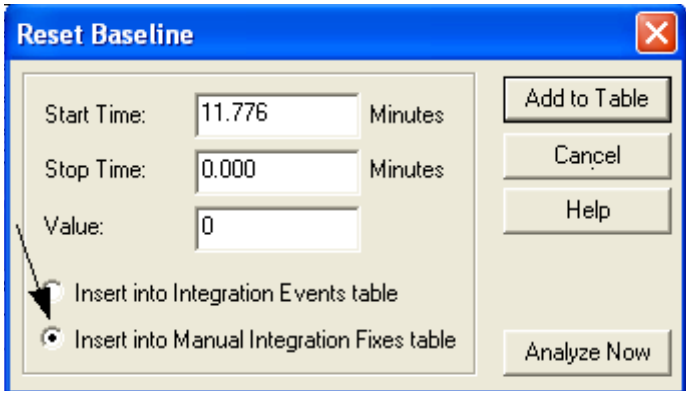
4. Open the data file by right clicking on the row of the run in the sequence. Choose OPEN DATA. The chromatograms for the two channels should appear.
5. Check the integrations for each target analyte.
6. If you need to do any integrations, the following are the common integration events:

Valley to Valley	***Draws the baseline to the minimum point between the peaks.***

Valley to Valley	***Draws the baseline to the minimum point between the peaks.***
Horizontal Baseline	***Draws the baseline horizontally determined by the beginning point in the range.***
Lowest Point Horizontal Baseline	*Draws a horizontal baseline determined by the lowest point in the range.*
Manual Baseline	*Draws a straight baseline from one point to another.*
Split Peak	*Draws a vertical line from the point to the baseline. It is commonly used to determine the end and beginning of a peak.*
Reset Baseline	*Draws the baseline up to a point on the chromatogram. It is commonly used to bring the baseline up due to a negative peak.*

After performing an event, a box will pop up and ask you to ANALYZE NOW.

Important! Before clicking ANALYZE NOW, make sure that you are inserting the event into the MANUAL Integration Fixes table. Inserting an event into the Integration Events table will add the event to the Method.



If you want to remove an integration event:

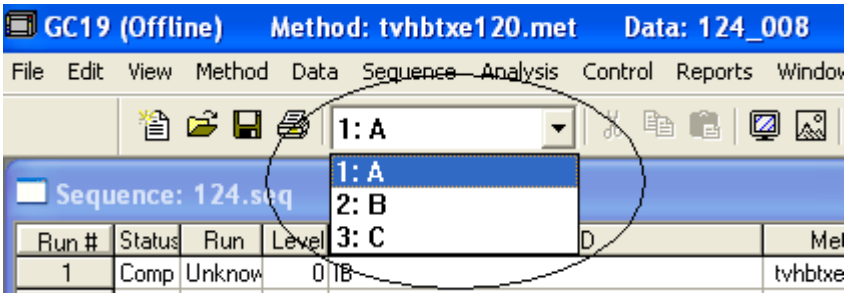
6.1 Open the Manual Integrations Fixes table by going to DATA > MANUAL INTEGRATIONS FIXES.

6.2 Select the integration you want to remove and press DELETE on your keyboard.

6.3 You will then need to analyze the data by going to ANALYSIS > ANALYZE.

To change what channel the table displays, select the channel from the drop down list on the main

toolbar, as shown below:



7. Repeat Steps 4 through 6 for the other data files. After you have finished any integration, you need to send the data to LIMS by printing the Method Custom Reports for the data to the ezchrom_capture printer.

   7.1 Go to SEQUENCE > PROCESS.

   7.2 Type the range of data you want to process in RANGE and make sure the box for PRINT METHOD REPORTS is checked.

   Note: Make sure that the printer configured to your EZChrom session is set to ezchrom_capture. You can check the printer by going to FILE > PRINT SETUP.

8. Some runs will need to be reprocessed with a different method (e.g. toxaphene###.met and/or chlordane###.met). To do this, change the method in the sequence table for those runs and save the sequence. Repeat Steps 3-7, now processing with the new method.

9. After EZChrom is finished processing the data, review the data in LIMS.

Making Compare-Plots ("Trace Overlays") in EZChrom:
If there is Toxaphene or Technical Chlordane in the samples, create a compare-plot to show the similarities of the sample pattern to that of a standard for that analyte.

1. Open the sample's data file in EZChrom.

2. Right click on the chromatogram from the channel you choose to use for the compare-plot and select PROPERTIES.

3. In the TRACE SETUP tab under Trace 1 Details (Current Data), change SCALE TO: to USER DEFINED. Change the values from Y MIN and Y MAX to leave enough space to add the CCV chromatogram(s).

4. At the top of the Data Graph Properties window, select the box under DATA SOURCE at row # 2. Click the right arrow and select OPEN DATA. Type the filename of the CCV data you will use for the compare-plot and click OPEN.

5. In the new trace's details, click the button to the right of TRACE to change the channel of the new trace to match the sample's chromatogram. Change the Y OFFSET to move the baseline of the new trace so that it does not overlap with the other chromatogram(s). You can also change the Y SCALE to size the chromatogram to better match the size of the sample's hit.

   Note: Choose CCV data from the same sequence and use the same channel for each of the chromatograms, so the retention times match.

6. To change the color and line-width of the chromatogram, go to the APPEARANCE tab in the Data Graph Properties window. Under ITEM, choose the chromatogram. With SUBITEM at TRACE, change the SIZE and COLOR.

7. Repeat Steps 3 through 6 for additional chromatograms.

   Note: You can click APPLY while you are changing values in the Data Graph Properties Window to see a preview of your changes.

8. Once you are finished adding traces and changing their appearance, click OK.

9. Send the chromatogram to LIMS by printing the compare-plot to ezchrom_capture. Change the print layout to Landscape by going to FILE > PRINT SETUP. Change the Orientation to LANDSCAPE then click OK. Right click the chromatogram and select UTILITIES > PRINT.

10. You can use the same added traces for other sample data chromatograms. When you open another data file in the same EZChrom session, the added traces should remain. You will just need to edit the chromatogram and select CLEAR OVERLAYS.

    Note: Remember to change the PRINT SETUP orientation back to PORTRAIT, otherwise the method reports will print in landscape mode.

Updating the Retention Times:

1. Create and enter a new method into the sequence before updating the retention times, then make sure the new method is currently open in your EZChrom session.

2. Open the data file you will use to update the retention times.

   Note: Retention times should be updated based on the middle calibration point.

3. Add the Retention Time Annotation to the chromatograms by right-clicking on any of the chromatograms and selecting ANNOTATIONS. Under AVAILABLE ANNOTATIONS, select RETENTION TIME and click the green right-arrow. Click APPLY TO ALL so that both chromatograms will display the retention times.

4. Open the peaks and groups table by going to METHOD > PEAKS/GROUPS. Using the retention times from the chromatograms, update the retention times under the column RET TIME. To change which channel is displayed, select the channel from the drop-down list on the main toolbar.

After you have changed the retention times on each channel, save the method by going to FILE > METHOD > SAVE.

APPENDIX_12: PROCESSING & REVIEWING DATA IN LIMS

Work up a sequence in LIMS.

1. Process sequence in EZChrom, making sure EZChrom print setup is set to \\LIMS\ezchrom_capture.

2. Open a web browser and go to the main Pesticides/PCB page.

3. Under "Recent Sequences", choose the GC and then choose the sequence you want to process.

   Note: The data must be processed through EZChrom and printed to ezchrom_capture before you can process it through LIMS. See Appendix_11 above for instructions on processing data through EZChrom.

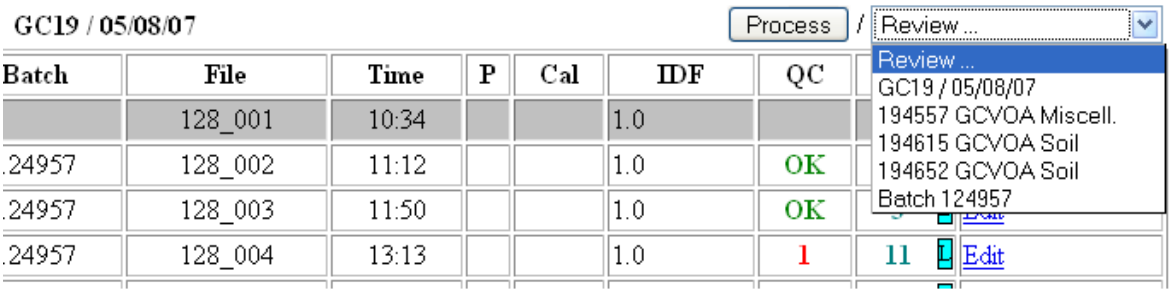
4. Check the sequence for any errors. LIMS takes the run information directly from the EZChrom sequence so any data entry error in the EZChrom sequence will affect the data processed by LIMS.

   If you need to make any changes to the sequence, click FIX at the lower right-hand corner of the LIMS sequence. Make the changes and click Update.

   To regenerate a LIMS user report for a specific run, check the box for the run and click PROCESS.

5. Once the sequence information is correct, use the 'Review App' to review the data, as described below.

6. In the top right, use the "Review" pull down menu and select your Sequence (for example: GC19A/ 05/08/07).



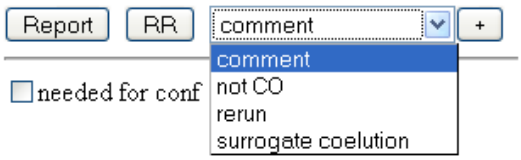
7. Two windows should open. The window on the left shows the main Review App frame (sequence, batch, ICAL) and the other shows the supporting documents (ie: EZChrom raw data reports, scanned prep logs). Clicking on an item in the left-hand frame will bring up the supporting documents for that item.

Important! You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

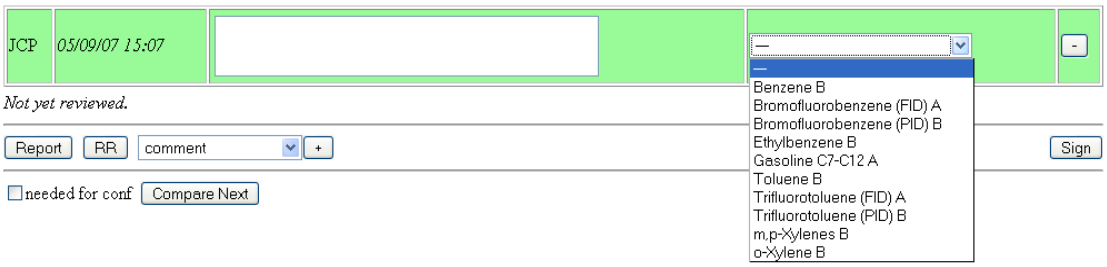
8. Check each run to ensure it is within the acceptable criteria, adding any necessary comments, and signing the data.

8.1 Scroll down the left screen and make sure the EZChrom TEH raw data and surrogate raw data reports match LIMS, and check for the proper chromatograms.

8.2 To add a general comment, click the (+) button while the pull down list to the left is set at COMMENT.



A comment box will appear with your initials.



Type your comment into the blank box.

If you want to associate the comment with a certain analyte in the run, you can choose that analyte on the pull down list to the right of the comment box.

If you want to delete the comment, click the (-) button.

If you need to edit a comment you made at an earlier time, click the (E) button.

Other choices under the Comment pull down list will add commonly used comments.

8.3 Mark data for re-analysis by clicking the 'RR' button. Any re-runs that are not because of linear range will require a brief comment as to why the data needs re-analysis. To undo an RR-flag, click the REPORT button.

8.4 For over linear range or over diluted samples, use the RR button with the rerun dropdown tab selected and type in the dilution factor needed. Finally, use the RX button for any re-extracts.

8.5 If the run is needed to confirm the results of another run, check the box 'NEED FOR CONF'.

8.6 Note that each analyte can only be 'chosen' once for a sample or QC. To choose analytes for reporting, check the box under the 'U' ("used") column; checking the box at the left of the 'U' header will choose all analytes for that run.

8.7 To change the reporting channel, check the channel for the analyte that needs to be changed or switch the whole list by clicking the CHANNEL pull-down list and choosing 'ALL A' or 'ALL B'. Click REFRESH to apply the changes.

9. Sign the data by clicking the SIGN button at the bottom right. Repeat the process with the other runs in your sequence.

Assemble a Batch in LIMS

Batch QC packages can be put together after the extraction lab has scanned in all the sample prep paper work, and all the QC samples (including the MSS and any necessary re-runs) have run and their brackets have been closed off with CCVs.

1. Use the C&T LIMS search function and type in batch number. This will find all data associated with the batch. Use the review apps pull-down menu and select batch number (ie: batch 121060).

2. Log in if necessary.

   Important! You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

3. In Review Application mode on left screen, the runs for the QC will appear along with associated ICALs, CCVs, and sequences. The associated CCVs should already be signed. If any are not signed, review and sign them.

4. Click the batch number at top of screen. Scanned documents should appear on right screen. Scroll down and check for any Corrective Action Records (CAR's) associated with the batch or with samples in the batch. Review and update the CAR.

5. Make sure that all extraction lab paperwork has been scanned correctly.

6. Review the sample prep log to make sure volumes, spike, and surrogate amounts are entered correctly in LIMS (data on left screen). Also look for any comments in the comment section, such as "2x surrogate" or "sediment".

7. Click on each QC data file and verify that all QC requirements (clean MB, analyte recovery, surrogate recovery, RPD) are met and that any necessary comments explaining recoveries that are outside of QC limits are present.

8. Check that the LIMS raw data numbers match the EZChrom data.

9. Sign each QC file, signifying that the data has been reviewed and is reportable or has been narrated if it is not being reported.

10. Verify that the run of the MSS that you are reporting is the same as the one that is linked to the MS/MSD. If the MSS was re-run for any reason, the run that we are reporting may not be linked correctly. If necessary, change the MSS run that is linked to the MS/MSD using the "MSS" link from the sequence where the MS/MSD ran. It must be changed for both the MS and the MSD individually. Be sure to make comments about any MS/MSD recovery or RPD failure.

11. Add any necessary comments to the batch as a whole using the comment tool at the bottom of the screen. For example, this would be where you could explain that the MS/MSD were not run if the MSS was run at a dilution >5x.

12. Finally, click the "Sign" button when the batch QC pack is complete and ready for review.

13. Notify a QC reviewer that it is ready (by instant message, telephone, or in person).

Assemble a Job in LIMS

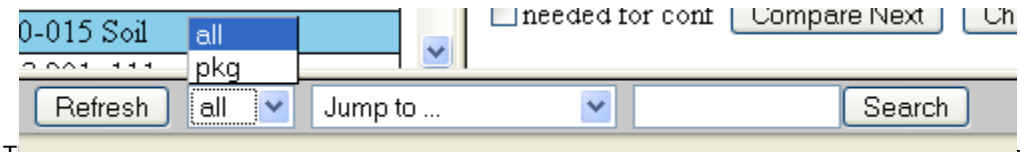
1. Review the job sheet. Check to make sure the correct cleaned-up extract of samples and QC has run and are ready to report. Make sure that any client-specific needs are addressed (for example, CCI needs to be on one instrument). Finally, search for any CARs associated with job and make any comments needed to complete the CAR.

2. Once you know what you need to report, begin by using the C&T search tool and type in job #, product (gcsv), and matrix (ie: 194505 gcsv soil). This will bring up sample data in LIMS. In the top right, use the "Review" pull down menu and select the job you would like to report.

3. Login if necessary.

   Important! You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

4. Go to the 'ALL" mode.



5. Make sure all of the samples in the job have run and the data is reportable. Sign and review all of the samples in the job and their associated batch QC. Make sure that the analytes you want to report have been 'chosen'.

6. Switch to PKG mode. Click the job number at the top left.

7. Click REPORTS. Check all the relevant samples and QC, then click PRINT to print all of the Form 1's on 2nd -page letterhead paper. Click DONE.

8. You may need to click REFRESH to see the Form-1 information on the Review App.

9. Check the Form-1 for any errors, missing analytes, and draft-flags.

10. Click the Job at the top of the list. Check the comments for any issues that you need to address, like missing signatures and documents.

11. Sign the job. Click CHECKLIST > PRINT to print the review checklist.

12. Complete and sign the checklist. Turn in the checklist and Form-1's to the QC Chemist, Group Leader, or Department Manager for review.

13. Remember to log out by clicking on your initials.

APPENDIX_13: EPA 608

The following criteria and discussion apply to any samples submitted for the EPA 608 analysis:

Initial Calibration:
Method 608 does not allow the use of single-point calibrations for Ar1221 through Ar1254. It only requires 3 points but requires that the RSD be ≤10% if average response is used; a regression curve may be used if the RSD exceeds 10% or whenever desired. Although 608 only requires 3 calibration points, the same general calibration criteria must be followed (ie: intermediate levels may not be dropped and the low point must be at or below the reporting limit). Any calibrations to be used for both 8081 and 608 must meet the 608 RSD requirement or use linear regression. Additional details regarding the procedure and SW-846 requirements are described in Appendix_9 above.

Calibration Verification (ICV/CCV):
Method 608 also only requires that a CCV (Continuing Calibration Verification) standard be analyzed once daily, however C&T policy is to follow the SW-846 guidance and analyze the CCV at the beginning of each sequence, every 12 hours, and at the end of the sequence. The %D for each analyte must be ≤ 15% and EPA 608 does not allow use of the "Grand Mean"; if the CCV does not meet this criterion, use the corrective action guidance described in Appendix_10 to determine the appropriate course of action.

Surrogates:
Method 608 does not discuss use of surrogate compounds, however C&T standard practice is to follow SW-846 guidance and use surrogates as a means of verifying the efficiency of the extraction and analysis. See Section 8 in the main body of this document for further guidance.

Batch QC Requirements:
Method 608 requires an LCS for every 10 samples, so any batch with more than 10 samples must include a BS and BSD. It also requires a matrix spike on 10% of samples submitted for the 608 analysis; if insufficient sample volume was submitted for matrix spikes, add a comment to this effect in the case narrative. EPA 608 defines the batch QC acceptance limits in Table 3 of the method; see the table below for these limits.

EPA 608- Batch QC Recovery Limits

Target Compound	Recovery Limits (%) *
Aroclor 1016	50 – 114
Aroclor 1221	15 – 178
Aroclor 1232	10 – 215
Aroclor 1242	39 – 150
Aroclor 1248	38 – 158
Aroclor 1254	29 – 131
Aroclor 1260	8 – 127
Surrogates	
Decachlorobiphenyl (DCB)	
Tetrachloro-m-xylene (TCMX)	

* *Method Note:* EPA 608 only discusses LCS and MS recoveries; it specifies the same limits for the two types of spike but does not discuss duplicates or surrogates and so does not define %RPD or surrogate recovery limits. These additional limits, along with recovery limits for the surrogates are determined by control charts.

Instrument Conditions:
Curtis & Tompkins uses current technology, namely capillary columns in place of the packed columns described in the method, so instrument conditions are somewhat different than those listed in the method. See Appendix 4 and instrument maintenance logs for instrument parameters.

Curtis & Tompkins, Ltd.

ICP METALS ANALYSIS

Inductively Coupled Plasma -
Atomic Emission Spectroscopy (ICP-AES)

Methods EPA 6010 & EPA 200.7

Approved by:

Porter Williams 3/9/12
Metals Group Leader / Date

3/9/12
QA Director/ Date

Read & Understood by:

3.09.12
Signature/ Date

3/23/12
Signature/ Date

3/9/12
Signature/ Date

Signature/ Date

3/22/12
Signature/ Date

Signature/ Date

Signature/ Date

Signature/ Date

Re-Approved by:

Signature/ Date

Signature/ Date

This SOP contains information that may not be disseminated to entities other than C&T staff, clients, and regulators.

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ICP METALS ANALYSIS

Methods EPA 6010 & EPA 200.7

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This SOP contains information that may not be disseminated to entities other than C&T staff, clients, and regulators.

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ICP METALS ANALYSIS

Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES)
Methods EPA 6010 & EPA 200.7

SCOPE

This document describes the analysis of waters, soils, and hazardous waste for inorganic elements by ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry) methods EPA 6010B, and EPA 200.7 with additional guidance provided by EPA 6010C. Methods 6010B and 6010C were written by the EPA's Office of Solid Waste with additional guidance for surface water and ground water, as opposed to EPA 200.7 which was written by the EPA's Office of Water for wastewater and drinking water. EPA 200.7 may also be requested for groundwater samples if the client is planning to discharge the water, with or without additional treatment, into a wastewater stream or into naturally occurring surface waters (bay or river). See Appendix_11 for a summary of EPA 200.7 requirements.

In this analysis, analytes in solution are nebulized and transported to a plasma torch. The emission spectra is then measured by spectrometry. Background subtraction is used to reduce interferences contributed by the plasma gas, reagents, and sample matrix. No digestion required for dissolved metals. Acid digestion prior to analysis is required for total metals in water, soil, and wastes.

See Appendix_4 for reporting limits and Appendix_10 for a discussion of interferences.

REFERENCES

Analytical Method:
EPA 6010B, Inductively Coupled Plasma AE Spectrometry, SW-846 Update 3, Dec.1996
EPA 6010C, Inductively Coupled Plasma AE Spectrometry, SW-846 Update 4, Feb 2007
EPA 200.7, Determination of Metals/Trace Elements in Water/Wastes by ICP-AES, Rev. 4.4

Sample Prep Methods:
EPA 3010A, Acid Digestion of Aqueous Samples, SW-846 Update 3, Dec.1996
EPA 3050B, Acid Digestions of Sediments, Sludges, and Soils, SW-846 Update 3, Dec.1996
EPA 200.7, Determination of Metals… by ICP-AES, Rev. 4.4

Additional SOP's and Guidance Documents:
NELAC Chapter 5, Quality Systems, June 2003
DoD Quality Systems Manual, Version 4.1, April 2009
DoE Quality Systems Manual, Version 2.2, Oct.2006
Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual, Rev 4.2 October 2010

C&T SOP QA 1.4, *Balance Calibration Check & Maintenance*
C&T SOP QA 1.5, *Calibrating & Maintaining Temperature Controls*
C&T SOP QA 1.6, *Pipet Calibration Check Procedures*

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C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL)*
C&T SOP QA 4.5, *Establishing Instrument Detection Limits (IDL)*
C&T SOP QA 8.4.1, *State Program Requirements*
C&T SOP QA 8.5.1, *DoD Program Requirements*
C&T QA SOP 8.6.1, *DoE Program Requirements*

PRESERVATION & HOLDING TIME

Preservation: HNO_3 to pH $\leq$ 2
Holding Time: 6 months

As of 4/11/07, 40CFR requires sample preservation to be performed within 15 minutes after collection of grab samples, composite samples, or aliquots taken from a composite taken automatically over time. For Dissolved Metals, the samples must be filtered and then preserved within 15 minutes of collection.

If a client requests that the lab filter the samples, a note to this effect should be placed in the case narrative. If the sample pH is >2 upon verification, EPA 200.8 requires that the sample be acidified to pH$\leq$2 *but* the lab must then wait 24 hours before verifying that the acidification was effective by rechecking the pH; if the pH was again >2, the process must be repeated.

SAFETY

Assume all samples, reagents and standards contain hazardous and/ or toxic material and take necessary precautions. Sample digests contain highly concentrated acids and should be handled with caution.

QC REQUIREMENTS

Project-specific quality assurance plans may have different criteria. If so, those requirements supersede this SOP for all samples related to that project.

a.) Initial Calibration:

An initial calibration (ICAL) curve consisting of a calibration blank and at least three standards must be established daily, prior to sample analysis. The lowest concentration standard must be at or below the reporting limit (see Appendix 4 for reporting limits) and the highest standard defines the top of the quantitation range. The correlation coefficient of this curve must be $\geq$ 0.995; if the calibration coefficient criterion is not met, the instrument must be recalibrated.

Note: for EPA 6010C the correlation coefficient of this curve must be $\geq$ 0.998; if the calibration coefficient criterion is not met, the instrument must be recalibrated.

b.) Calibration Verification:

An Initial Calibration Verification (ICV) standard obtained from a second supplier and Initial Calibration Blank (ICB) verification must be run at the beginning of each analytical run. A

This SOP contains information that may not be disseminated to entities other than C&T staff, clients, and regulators.

Continuing Calibration Verification (CCV) standard and Continuing Calibration Blank (CCB) verification must be analyzed after every 10 analytical samples, including batch QC samples, and at the end of the sequence.

The concentration of the CCV is set at the midlevel of the calibration curve , with a tolerance of ± 10% of its true value for EPA 6010B and 6010C analysis and ± 5% for EPA 200.7. If any element falls outside these limits, all samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing CCV only if the sample result is determined to be less than the specified RL.

For EPA 6010C analysis: In addition to the midlevel ICV and CCV, a low level ICV should be run following initial calibration. The LLICV should be prepared at the limit of quantification (LOQ) for the calibration with a tolerance of ± 30% of its true value. Both the LLICV should be prepared from the same source as the calibration standards. If any element falls outside these limits, all samples affected by the failure must be reanalyzed.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the LLICV must be within ± 20% of its true value, unless there is a variance where the LLICV must be within ± 30%.

Note: The USACE recommends that an ICAL standard, or a standard from the same manufacturer as the ICAL standards, be used for the CCVs, to more readily identify problems that are due to changing instrument conditions and are not due to differences between standards.

c.) Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB):
An initial calibration blank (ICB) verification must be run at the beginning of each analytical run, with the ICV. A continuing calibration blank (CCB) verification must be analyzed (with each bracketing CCV), after every 10 analytical samples, including batch QC samples, and at the end of the sequence. Target elements should not be detected in the CCB at any level above 3 times the Instrument Detection Limit (IDL).

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the ICB/CCB must be ND > LOD

d.) Interference Check Standard A (ICSA):
An interference check standard (ICS-A) containing only common interferents standard should be analyzed at the beginning of each sequence, after the calibration standards and verifications, to demonstrate that high levels of interferents are not significantly biasing sample results, in either a positive or negative fashion. The determined concentration of the non-interferent should be no more than ±RL in either direction.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the measured concentrations of any elements that are not included in the ICS-A standard must be ND > LOD

e.) Interference Check Standard A-B (ICSAB):

An interference check standard (ICS-AB) containing both common interferents and low-level target analytes standard should be analyzed at the beginning of each sequence, after the calibration standards and verifications, to demonstrate that the interference corrections are effective and are correctly applied. The ICS-AB should also be analyzed at the end of the sequence to demonstrate that instrument conditions have not significantly changed over the course of the sequence - see *Note* below. The ICS-AB standard should contain interferent elements at concentrations greater than 100 ppm and the elements of interest at concentrations between 0.5 to 1.0 ppm.

The recovery for each element should be within 80-120% of the true value or subsequent sample results for that element cannot be reported.

Note: Although EPA 6010B does not require analysis of an ICS-AB at the end of the sequence, most Department of Defense (Army, Navy, etc) project plans include this requirement, so it is always a good idea to close the sequence with this standard.

f.) Batch QC:

The following quality control (QC) samples must be prepared in the same manner as the analytical samples at a rate of once per twenty or less samples for method 6010 and once per ten or less samples for method 200.7. C&T in-house acceptance limits are updated semi-annually; based on control charts of the previous year's data. See the associated SOP '*6010B QC Limits, Table-1'* for the current limits. For EPA 200.7, the QC recovery limits are specified in the method and can be found in Appendix 11.

Note: Project-specific quality assurance plans may have different criteria. If so, those requirements supersede this SOP for all samples related to that project.

Method Blank (BLANK): The purpose of the method blank is to ensure that the digestion and analysis process does not in any way contaminate the analytical samples. Deionized water is carried through the entire digestion process and analyzed. The results for the preparation blank should be <1/2 RL and must be < RL for all target elements. If the sample result for that compound(s) is greater than ten (10) times the amount found in the method blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis. If the sample result for that compound(s) is greater than the reporting limit but less than ten (10) times the amount found in the associated method blank, the samples must be re-digested and reanalyzed

EPA 200.7 Method Note: Method 200.7 requires redigestion and reanalysis of any sample associated with a method blank containing laboratory contamination greater than 2.2x the MDL for that element and <10x the concentration in the sample. If the sample is ND for the blank contaminant or is >10x the level found in the blank, the sample may be reported without corrective action.

This SOP contains information that may not be disseminated to entities other than C&T staff, clients, and regulators.

Blank Spike (BS) and Blank Spike Duplicate (BSD): The purpose of the blank spikes is to demonstrate that the sample preparation and analysis procedures are accurate (recovery) and precise (RPD) in the absence of matrix interferences. A known concentration of each element is added to deionized water and carried through the entire digestion and analysis process.

For EPA 6010, the recoveries and RPD should fall within C&T in-house limits, or the samples associated with it should be redigested and reanalyzed. For EPA 200.7, the recoveries must fall within 85-115%.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD): The purpose of the matrix spikes is to demonstrate that the sample preparation and analysis procedures are accurate (recovery) and precise (RPD) in the possible presence of matrix interferences. A known concentration of each element is added to a real-world sample and carried through the entire digestion and analysis process.

For EPA 6010, the recoveries and RPD should fall within inhouse acceptance limits. For EPA 200.7, the recoveries should fall within 70-130%. If recovery limits are not met, an acceptable explanation and narration, or redigestion of samples is required. If the concentration of any element in the spiked sample is greater than four times the spiking level, the recovery is considered 'Not Meaningful' and LIMS will place an "NM" flag on the report.

Sample Duplicate (SDUP): For leachates or other samples known to contain high levels of target analytes, a sample duplicate and sample spike may be analyzed in place of the MS/MSD. The selected sample is prepared and analyzed in duplicate to determine the precision of the sample preparation and analysis process in the presence of potential matrix interferences. If the RPD exceeds C&T RPD limits for a majority of the analytes of interest, the source of the error must be identified and narrated or the affected samples redigested.

Sample Spike (SSPIKE): A second aliquot of a sample is spiked with a known concentration of elements to determine the accuracy of the sample preparation and analytical process in the presence of potential matrix interferences. If C&T recovery limits are not met, an acceptable explanation and narration, or redigestion of samples is required. If the concentration of any element in the spiked sample is greater than four times the spiking level, the recovery is considered 'Not Meaningful' and LIMS will place an "NM" flag on the report.

g.) Sample Interference Verifications:

The method recommends that whenever a new or unusual matrix is encountered, a series of tests be performed to ensure that neither positive nor negative interferences are distorting the accuracy of the reported values. These are optional steps that should be implemented for any batch containing "Level 3" or Level 4" samples, as nearly all Department of Defense

project plans include this requirement. These should also be analyzed whenever the analyst suspects that sample viscosity, salt content, or other matrix interferences are likely.

Serial Dilutions: Analysis of a 5x dilution should agree within ± 10% (90-110% recovery) of the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference should be suspected.

EPA 200.7 method note: Serial Dilution recovery limits are 95-105%.

Post Digest Spikes: An analyte spike added to a portion of a prepared sample digest, or its dilution, should be recovered to within 75-125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits a matrix effect should be suspected.

EPA 200.7 method note: Post Digestion Spike recovery limits are 90-110%
EPA 6010C method note: Post Digestion Spike recovery limits are 80-120%.

h.) Multi-Spectral Fitting (MSF):
MSF is a method of correcting for spectral overlap of interferents with the analyte of interest by collecting spectra of a blank, analyte, and individual solutions of each interferent. In MSF models, all components are assumed to be linearly independent and the MSF is then independent of concentration or plasma effects. This method cannot be used if the spectra of two analytes directly overlap, but is the preferred method for those analytes whose spectra are even slightly offset.

i.) Inter-Element correction (IEC) factors:
An IEC is a method of correcting for the spectral overlap of high levels of interfering elements, upon the analyte of interest, in those instances where Multi-Spectral Fitting cannot be applied. Mathematical correction factors are applied to the emission intensities. IEC's are limited in that the interfering element must be within the linear range and the plasma conditions must not be changed. The IEC's are determined through analysis of single-element standards and examination of the resulting data against the intensities collected at the remaining wavelengths to determine what other elements may be affected by a high level of that element. Any IEC's must be applied where needed and updated every six months or when an instrument change occurs in the torch, nebulizer, injector, optics, or plasma condition.

j.) Method Detection Limit (MDL):
MDL studies are performed when major changes to instruments or methods occur, in order to demonstrate that the sample preparation and analysis procedures are adequate to meet required reporting limits. The MDL is determined by digesting and analyzing at least 7 replicates of a low-level blank spike, determining the standard deviation for each element, and applying a multiplier. See the QA SOP "Determining MDLs" for details.

k.) Instrument Detection Limit (IDL):
The DOD-QSM and EPA 200.7 require that IDL studies be performed quarterly for each instrument. Because method EPA 6010B does not discuss IDL's, C&T follows the procedure described in EPA 6020 Section 8.2 and determines the IDL by analyzing seven instrument blanks (that have *not* been digested), on each of three non-consecutive days. The standard deviation of the calculated concentrations is determined for each day; the IDL is the average of the three standard deviations. The IDL must be less than the water MDL for that instrument.

l) **Limit of Detection (LOD)** must be determined quarterly and is based on a laboratory control sample (MDL check sample) that is spiked 2 to 4 times the MDL, digested and analyzed on every instrument. See the QA SOP for Method Detection Limits for details.

m.) **Limit of Quantitation (LOQ)** must be determined quarterly and is based on a laboratory control sample that is spiked 1 to 2 times the reporting limit. It is only analyzed once per method. See the QA SOP for Limit of Quantitation for details.

EQUIPMENT
MET-08: Perkin-Elmer ICP, Model 5300 DV with CETAC autosampler
MET-09: Perkin-Elmer ICP, Model 5300 DV with AS93Plus autosampler
WinLab 32, Perkin-Elmer Data Acquisition Software, Version 3.0

DAILY MAINTENANCE
- Replenish the rinse-water reservoir daily with 2% HNO_3 - 2% HCl.
- Change the pump windings for the internal standard solution every day or two.
- Change the sample tubing every 1 to 2 weeks, or whenever bubbles appear in the lines.

DAILY INSTRUMENT SEQUENCE
1) Turn the ICP on and allow the instrument to stabilize for about 30 minutes, or longer (up to 90 minutes) when the pump windings have been changed. See Appendix 8 for instrument start-up procedures.

2) Verify that the lamp is correctly aligned, as described in Appendix 8.

3) Calibrate the ICP by running a calibration blank followed by at least three calibration standards, in increasing order of concentration, at levels that bracket the quantitation range;

the lowest standard must be at or below the reporting limit and the highest standard determines the upper end of the quantitation range (see Appendix_3).

4) Send the calibration data files to LIMS, as described in Appendix_8.

5) Review the ICAL summary to determine if the sequence can be continued:

- The percent RSD should be ≤ 5% for the three replicate exposures of each standard. See Appendix_8 for software instructions.

  EPA 200.7 note: The RSD limit is ≤ 3%.

- The correlation coefficient for each element must be ≥ 0.995.

  EPA 6010C note: The correlation coefficient must be ≥ 0.998.

- The highest concentration standard may be omitted so as long as there are at least three points remaining and the remaining highest point defines the top of the calibration range (any digests which exceed this concentration must be diluted and reanalyzed).

- The lowest concentration standard may be omitted from curve if, *and only if*, the resulting lowest standard is at or below the reporting limit for samples and there are at least three points remaining.

- Mid-point standards may not be omitted simply to improve the correlation coefficient. They may, however, be reanalyzed if poor aspiration is suspected. The reanalysis must occur immediately after the curve so long as no sample digests were analyzed since the last calibration standard and all elements are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular element.

6) Verify the calibration curve by running an ICV standard (obtained from a different manufacturer from the calibration standard) for all elements of interest. The ICV %D's must fall within acceptance limits for the elements of interest before analysis can proceed. See Appendix_3 for the concentrations in this standard.

   6010 Limits: 90-110% recovery
   200.7 Limits: 95-105% recovery

7) **For EPA 6010C analysis:** Analyze a low level ICV (LLICV) after running the initial calibration. The LLICV should be prepared at the limit of quantification (LOQ) for the calibration with a tolerance of ± 30% of its true value. The LLICV should be prepared from the same source as the calibration standards. If any element falls outside these limits, all samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing LLICV only if the sample result is determined to be less than the

specified RL An element may also be reported in the event of a failing LLICV if the LOQ used for the sample result is greater than or equal to the level of a passing midlevel CCV or a passing LLICV run at a higher concentration.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the LLICV must be within ± 20% of its true value.

8) Analyze an Instrument Calibration Blank (ICB), consisting of deionized water acidified with 5% HCl and 5% HNO_3. Target elements should not be detected in the ICB at any level above 3 times the Instrument Detection Limit (IDL).

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the ICB/CCB must be ND > LOD

9) Analyze the ICS-A to demonstrate that high levels of interferents are not biasing low-level quantitation. The determined concentration of the non-interferent should be ≤RL in either direction (+/-), except for those elements that are considered by the manufacturer to be 'trace' contaminants of the high level elements and are listed on the Certificate of Analysis. If this standard fails acceptance criteria, check the interference corrections (MSF); update the MSF if necessary and reprocess the sequence to that point.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the measured concentrations of any elements that are not included in the ICS-A standard must be ND > LOD.

10) Analyze the ICS-AB to demonstrate that quantitation in the presence of high-level interferents is acceptably accurate. The results for the non-interferent elements in the ICSAB must be within ± 20% of the true values (80-120% recovery). If this standard does not meet the acceptance criteria, the analysis must be terminated, the method corrected for the interference, and the analysis restarted from calibration.

11) Now after all of this QC, real samples may be run. Collect the digestates, job sheets, LIMS prep sheet, and copy of the benchbook page from the prep chemists. Sign the "Received by" line on the LIMS prep sheet to maintain internal chain-of-custody of the digestates.

12) Decant the digestates into labeled autosampler tubes.

Note: Analyze leachates at a 10x dilution so the high salt content of the leaching fluid will not 'salt out' in the aspirator and clog the injection system.

13) Choose a sample for the serial dilution and post-digestion spike; this sample is usually the same sample used for the MS/MSD, but may be different if requested by a client.

12.1) Prepare the Serial Dilution by measuring 2.0 mL of the sample digestate into a disposable centrifuge tube labeled with the sample number and "SD". Add 8.0 mL of acidified (5%HCl, 5%HNO_3) deionized water. Cap and invert several times to mix.

Enter the serial dilution into the LIMS 'Prep Entry' table, using the same mass:volume as the original sample. In the instrument sequence, enter it using the stype 'SER' (like 'BS'), with an instrument dilution factor of 5.

12.2) Prepare the Post-Digestion Spike by measuring 10.0 mL of the sample digestate into a disposable centrifuge tube labeled with the sample number and "PDS". Add 0.10mL of the same spike used for the MS/MSD. Cap and invert several times to mix.

Enter the post-digestion spike into the LIMS 'Prep Entry' table, using the same mass:volume as the original sample. In the sequence, enter it using the stype 'PDS' along with the LIMS S# of the spiking standard used, and a dilution factor of 100 for the spiking standard.

14) After ten samples are analyzed, and at the end of the analytical run, the calibration curve must be re-verified by running a Continuing Calibration Verification (CCV) and a Continuing Calibration Blank (CCB). Note that the ICS-A and ICS-AB are considered analytical samples in the first group of ten samples.

See Appendix_8 for instructions on setting up an autosampler sequence. A typical analytical sequence is shown below:

Calibration Blank
Calibration Standard, low level
Calibration Standard, mid-level
Calibration Standard, high level
Instrument Blank
Initial Calibration Verification (ICV)
Initial Calibration Blank (ICB)
Interference Check Standard A (ICSA)
Interference Check Standard A+B (ICSAB)
Method Blank
Blank Spike
Blank Spike Duplicate
Sample Duplicate
Sample Spike
.. 3 more samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
.. 10 samples ..
Continuing Calibration Verification Standard (CCV)

Continuing Calibration Blank (CCB)
.. 10 samples ..
Interference Check Standard A+B (ICSAB)
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)

The sequence must end with a CCV and CCB, regardless of the number of samples that have been analyzed.

Note: Although EPA 6010B does not require analysis of an ICS-AB at the end of the sequence, EPA 200.7 and most Department of Defense (Army, Navy, etc) project plans include this requirement, so it is always a good idea to close the sequence with this standard. If an element does not fall within 80% - 120% all samples analyzed for that element must be reanalyzed.

QUANTITATIVE ANALYSIS

The Perkin-Elmer ICP automatically adds internal standard (Yttrium), which helps compensate for viscosity and transport interferences. The sample is then transported through the nebulizer and vaporized in the plasma. The spectrometer measures the changing intensity of the selected wavelength either radially (across the radius of the plasma) or axially (along the radius of the plasma). For all samples and standards, the instrument collects information for three exposures and reports the average intensity of these exposures, along with the %RSD between the exposures.

Quantitation is based on comparison of the intensity of the target element and internal standard to the initial calibration curve for that element, with adjustments for the sample preparation concentration factor and instrument dilution factor. The intensity reading on the Perkin-Elmer software hardcopy report is the “corrected” intensity that has already been adjusted for the internal standard response; see Appendix_8 for instructions to view or print the raw, uncorrected intensities. See Appendix_1 for example calculations. Concentrations are expressed as micrograms per liter (µg/L) or milligrams per kilogram (mg/kg).

All results are reported on a wet-weight (“as received”) basis unless otherwise requested by the client. If the client requests ‘dry-weight’ corrections, the ‘wet-weight’ results in the results database are corrected for moisture by LIMS when producing the final report forms.

1.) Evaluate the Internal Standard Recoveries

The Yttrium internal standard recovery should be between 30-200% for all standards and samples, or that data should be considered suspect and those standards or digestates rerun. If the affected run is a CCV or CCB, all samples bracketed by that run should also be rerun.

Note: The 30-200% limits specified above are C&T’s in-house limits, as EPA 6010B does not specify internal standard recovery limits.

2.) Evaluate the CCV's

Target elements must not be detected in the calibration blank at any level greater than the reporting limit. The concentration of the CCV must be varied within the calibration range, over the course of the sequence, with recoveries between 90-110% (or 95-105% for EPA 200.7). If the %D for any element is outside this acceptance window, LIMS will use the following to determine if the associated results are reportable:

- If the failing element is not a target analyte for the associated samples, sample results should be reported without reanalysis.
- If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.
- If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.
- If the compound fails the %D criterion due to a low response, the sample must be reanalyzed as the low bias may result in false negatives or misquantitation.

Reported sample and batch QC results must be bracketed by acceptable calibration verification standards and blanks.

3.) For EPA Method 6010C, Evaluate the LLCCV

Analyze a low level CCV (LLCCV) following the ICV. The LLCCV should be prepared at the limit of quantification (LOQ) for the calibration with a tolerance of ± 30% of its true value. The LLCCV should be prepared from the same source as the calibration standards. If any element falls outside these limits, all samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing LLCCV only if the sample result is determined to be less than the specified RL An element may also be reported in the event of a failing LLCCV if the LOQ used for the sample result is greater than or equal to the level of a passing midlevel CCV or a passing LLCCV run at a higher concentration.

Note: For any Department of Defense (Navy, USACE, AFCEE) project that references the DoD Quality Systems Manual (QSM), the LLCCV must be within ± 20% of its true value.

4.) Evaluate the Batch QC Results

- **Prep Blank:** The results for the preparation blank should be <1/2RL and must be <RL for all target elements. If reanalysis confirms the contamination, use the following steps to determine if the sample results may be reported:

a. If the concentration of the contaminant is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

b. If the target element(s) found in the blank was not detected in the associated samples, the data may be reported and the problem narrated.

c. If the target element(s) found the method blank was also detected in the associated samples, but the level in the samples is greater than 10x the level in the blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

d. If the target element(s) detected in the blank were also detected in the associated samples, but at levels less than 10x the level in the blank, and reanalysis confirms the problem, the samples containing the contaminant must be re-batched and reanalyzed. Initiate a Corrective Action Report (CAR) immediately so that re-digestion can begin within the clients requested turn-around time, if necessary.

Note: EPA 200.7 requires redigestion and reanalysis of any sample associated with a method blank containing laboratory contamination greater than **2.2x the MDL** for that element and <10x the concentration in the sample. If the sample is ND for the blank contaminant or is >10x the level found in the blank, the sample may be reported without corrective action.

- **Blank Spike (BS) and Blank Spike Duplicate (BSD):** The recoveries and RPD should fall within acceptance limits, or the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine if the sample results may be reported:

a. If the samples are being analyzed for only a subset of the target element list (ie: lead only, LUFT 5, etc.) and those elements all pass acceptance criteria, the data may be reported without further corrective action.

b. If a high recovery is observed but that element was not detected in the associated samples, note the failure on the Data Review Checklist and report the data without re-digestion, as the potential high bias does not affect the sample results.

c. If a high recovery is observed and the samples contain that element at levels above the reporting limits, the samples containing that element must be re-digested.

d. If a high RPD is observed but the recoveries are within acceptance limits and the samples do not contain that element, note the failure on the Data Review Checklist and report the data without re-digestion, as the lack of good precision data does not affect ND samples.

e. If a high RPD is observed and the samples contain that element at levels above the reporting limits, those samples containing that element must be re-extracted.

- **Matrix QC (SSPIKE/SDUP or MS/MSD):** The recoveries and RPD should fall within acceptance limits, or the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine any necessary corrective action:

  a. If the concentration of a target element in the sample is greater than the linear range and the sample needs to be rerun for just that compound, report the MS/MSD with a LIMS-flag of ">LR" on those recoveries, without reanalysis.

  b. If the concentration of a target element in the sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a ">LR" flag to those recoveries. Report the data without reanalysis.

  c. If the concentration of a target element is greater than 4x the spiking level, LIMS will apply a "NM" (for "Not Meaningful") flag to those recoveries. Report the data without reanalysis.

     Note: If the concentration of a target compound is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

  d. If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

  e. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

- **Serial Dilutions:** Analysis of a 5x dilution should agree within 90-110% recovery (95-105% for EPA 200.7) of the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference, such as viscosity of the digestate, should be suspected. If for a given project, all elements repeatedly show an increased response in the Serial Dilution, when compared to the initial run, discuss the problem with the Department Manager and/or the Project Manager for that account, as the samples may require routine dilution to overcome viscosity effects.

- **Post Digest Spikes:** An analyte spike added to a portion of a prepared sample digest, or its dilution, should be recovered to within 75-125% (80 – 120% for EPA 6010C or 90-110% for EPA 200.7) of the expected concentration. The spike addition should produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. If the recoveries of all elements show a uniform bias in either direction, make and analyze a new spike to verify that it was prepared correctly. If the spike is not

recovered within the specified limits a matrix effect should be suspected and the sample should be diluted (to dilute out the matrix interference).

5.) Evaluate the Sample Results

Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

Examine the sample results to verify that all results are within the linear range. If the concentration of any requested target element is greater than the highest calibration standard for that element, use volumetric pipettes to prepare a dilution of the digestate so that the highest target element is in the upper half of the calibration range; see Appendix_2 for instructions. If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

Examine the sample results to verify that the RSD between exposures is less than 20%. Any sample result with a duplicate exposure RSD greater than 20% must be reanalyzed. If the RSD is still greater than 20%, report the exposure with the lower %RSD. Any sample with requested element concentrations above the linear range must be diluted and reanalyzed.

6.) Reviewing and Reporting Results

1.) Open a web browser and go to the main METALS page.

2.) Under Recent Sequences, choose the instrument and then choose the sequence you want to review.

3.) Check the sequence for any errors. LIMS takes the run information directly from what is typed in the sequence. Typing errors can affect how the data is processed by LIMS.

If you need to make any changes to the sequence, click FIX. Make any needed changes and click UPDATE.

4.) Once everything is correctly processed in the sequence, review the sequence to see that all samples, STDs, and QC meet acceptance criteria. Samples and QC should have an “OK” flag in the QC column of the sequence, otherwise reanalysis may be required. Once the sequence has been reviewed, it must be signed in the REVIEW APP.

5.) Open REVIEW APP for the sequence by choosing the sequence (e.g. MET14 / 05/08/07) under the Review pull down list on the top right corner of the sequence page.

6.) Two windows should open. One window shows the main Review App page. One window shows the documents (e.g. reports, scanned documents, Form 1s, etc).

IMPORTANT! Make sure you are logged in with your own user initials. The current user's initials are displayed on the bottom left corner of the review app window. To logout, click the user initials and Review App will ask you to login.

7.) The sequence is listed on the left frame of the Review App page. Clicking on an item on that list will display the LIMS report and any associated documents.

8.) The analyst adds a comment that the sequence has been verified/reviewed for either all files in the sequence or those files the analyst has reviewed if the sequence is still running.

9.) From another open web browser go back to the main METALS page and click on run chooser.

10.) Enter the login number, analysis or rgroup (metals), and matrix for the job to be reported. Choose the result(s) to be reported for each sample and QC for the job.

11.) Select reports from the top left side of the screen. From the views section in the upper right select possible. This will list the possible reports that can be printed. Select the reports to be printed and print to nowhere. This will select and lock the final results in the LIMS database.

12.) From the REVIEW APP page you left open previously, enter the batch number in the box in front of the search button and click the button.

13.) Review the results and sign the main page of the batch.

14.) From a different open web browser, go back to the main METALS page and enter the job number you wish to report in the search box and select search. Open REVIEW APP for the job by choosing the job (e.g. 214373 METALS Filtrate) under the Review pull down list on the top right corner of the page.

15.) Review the results and sign the main page for the job.

16.) Inform the group leader that the job is ready for review.

17.) When the reviewer or group leader does the final review of the job, the level III or IV packages are generated at that time.

SAMPLE PREP DOCUMENTATION

A copy of the digestion benchbook page for the sample digestion must be scanned in under the batch number and included with the reported data. The digestion benchbook entries should include:

- C&T sample ID's and unique container identifier,
- date of sample digestion, initial volume or weight of sample, and final digestate volume,
- identity of QC samples (spikes, duplicates & LCS),
- amount of spikes added and LIMS identification numbers of all spiking solutions,
- a list of all reagents used (C&T ID or manufacturer and lot number),
- indication of whether or not the digests were filtered after digestion,
- any unusual occurrences observed during the digestion procedure

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POLLUTION PREVENTION
Prepare only sufficient standard and reagent volume to use within the shelf-life of the standard to reduce the volume of waste generated by the laboratory.

WASTE DISPOSAL
All digests are kept for at least 6 months prior to disposal; after 6 months, the digests are included in the 'Corrosives' waste stream. Instrument waste is collected in a polyethylene carboy and discarded into the 'Corrosives' waste stream.

REVISION HISTORY
This is rev 14. Rev 13 was reviewed and changed as follows:

- Remove: **Analyze a low level ICV (LLICV) ~~at the end of each batch or sequence~~.**

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APPENDIX_1: CALCULATIONS

Sample Concentration via Linear Regression with Internal Standard
Concentration (ug/L or ug/Kg) = (a0 + a1 * Ax)) * PDF * IDF

Where:
a0 = Y-intercept of regression equation
a1 = slope of regression equation
Ax = (IS-Corrected*) Intensity reading for the analyte in the sample, or (Sample Intensity / Internal Standard Intensity)
IDF = Instrument Dilution Factor
PDF = Prep Dilution Factor (Vf/Vi or Vf/Wi),
Where Vf = digestate final volume,
Vi = initial volume of liquid sample
Wi = initial weight of solid sample

Moisture Corrected Results
Dry Weight Concentration (ug/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

BATCH QC CALCULATIONS

Percent Recovery (%R):
The recovery is the measured concentration divided by the true concentration of the spike.

%Recovery = (Cf – Cs) / (Cws * Vws/ S) *100

Where:
Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard
S = Sample weight or volume

Relative Percent Difference (RPD):
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

%RPD = |(Cs - Cdup)| / ((Cs + Cdup)/2) * 100

Where:
Cs = measured sample concentration
Cdup = measured concentration in the duplicate

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For soil MS/MSD's where the sample weights are not weight-targetted, the expected concentrations will vary with sample weight (because the same volume of spike standard is being added to different weights of sample) and must be accounted for when calculating RPD:

$$\%RPD = |((Wms/Wmsd)*Cms - Cmsd)| / (((Wms/Wmsd) * Cms + Cmsd)/2) * 100$$

CALIBRATION EQUATIONS

Linear Regression Equations

$$y = mx + b$$

Where: y = response (Ax for external standard, or Ax/Ais for internal standard)
Where Ax = Area of compound
Ax / Ais = Area of compound divided by area of internal std
x = concentration (Cx for external standard, or Cx/Cis for internal standard)
m = slope
b = intercept

$$\text{Slope (m)} = [(\sum wx_i y_i * \sum w) - (\sum wx_i * \sum wy_i)] / [(\sum w * \sum wx_i^2) - (\sum wx_i * \sum wx_i)]$$

$$\text{Intercept (b)} = y_{avg} - (m * x_{avg})$$

Correlation Coefficient (r)

$$r = \frac{[(\sum w * \sum wx_i y_i) - (\sum wx_i * \sum wy_i)]}{\sqrt{\{[(\sum w * \sum wx_i^2) - (\sum wx * \sum wx_i)] * [(\sum w * \sum wy_i^2) - (\sum wy_i * \sum wy_i)]\}}}$$

Coefficient of Determination (r^2) = r * r

Where: x_i = individual values for the independent variable (concentration)
y_i = individual values for the dependent variable (response, area)
w = weighting factor (for no weighting w = 1)
x_{avg} = average of the x-values
y_{avg} = average of the x-values

Make a Working Standard from a Source (Stock) Standard:
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where: Vss = Volume of Source Standard (mL) needed to make Working Standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

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APPENDIX_2: SAMPLE DILUTIONS

If the sample concentration is greater than the highest point in the calibration curve, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range. Examine the original sample data to determine what dilution factor will be required to bring the absorbance down to the middle of the calibration range.

Using calibrated auto-pipettes, dilute the following volumes of sample digestate in a disposable centrifuge tube; adjust the acid concentration to 5%. Cap and invert three times to mix, allowing sufficient time for complete mixing with each inversion.

DILUTIONS

Instrument Dilution Factor (IDF)	Digestate Volume	Add Volume (mL) Acidified DI Water	Final Volume (mL)
2	5.0 mL	5.0	10
5	2.0 mL	8.0	10
10	1.0 mL	9.0	10
20	0.50 mL	9.5	10
50	0.20 mL	9.8	10
100	0.10 mL	9.9	10

If a sample should need a dilution of more than 100x, prepare a 100x dilution first, then use that to make subsequent dilutions.

SERIAL DILUTIONS

Instrument Dilution Factor	Using Primary Dilution	Volume of Primary (100x) Dilution	Add Volume (mL) Acidified DI Water	Final Volume (mL)
500	100x	2.0 mL	8.0	10
1,000	100x	1.0 mL	9.0	10
2,000	100x	0.5 mL	9.5	10
5,000	100x	0.2 mL	9.8	10
10,000	100x	0.1 mL	9.9	10

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APPENDIX_3: REAGENTS & STANDARDS

REAGENTS

The preparation of all liquid or solid reagents, including dilutions into DI water, must be documented in the reagent prep benchbook. Each reagent is assigned a unique ID, based on the manufacturer and the date prepared. This ID is then recorded in the digestion benchbook each time the reagent is used.

Label each reagent with the reagent ID, concentration, prep date, and expiration date. All reagents should be prepared and stored in freshly cleaned glassware. Expired, discolored, or contaminated reagents should be discarded and the bottle cleaned before reuse.

Argon, Purity: 99.99%
Air, C&T house compressed air

Deionized water, ASTM Type II (ASTM D1193)

Aqua Regia: *prepare daily*
Prepare immediately before use in a glass bottle by adding 3 volumes of concentrated HCl to one volume HNO_3, typically preparing about 125mL aqua regia per batch (32mL HCl + 96mL HNO_3 => 128mL total). Aqua Regia must be prepared daily.

Nitric Acid (HNO_3), concentrated, InstraAnalyzed grade
JT Baker catalog # 7697-37-2
Store unopened bottles in the corrosives cabinet and open bottles under the fume hood for up to ten years.

Hydrochloric Acid (HCl), concentrated, InstraAnalyzed grade
JT Baker catalog # 7647-01-0
Store unopened bottles in the corrosives cabinet and open bottles under the fume hood for up to ten years.

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STANDARDS

All source standards must be documented in LIMS upon receipt, through the Standards Menu". The LIMS S-name is unique to the vendor that the source is obtained from; if a source standard is obtained from a different vendor, a new S-name must be assigned and the information entered in the "Standard Entry" table before the standard can be assigned a unique S# (standard number).

The LIMS Standard Maintenance database includes the catalog#, lot#, expiration date, and concentration of the standards as they are received from the vendor. Write the S# and the date received on both the standard vial and on the 'Certificate of Analysis' that accompanied the standard. If the supplier did not provide a certificate, call and request that a copy be faxed. The Certificate of Analysis must be kept on file in the appropriate binder.

Prepare working standards by diluting source standards to volume in a Class-A volumetric flask. Document the preparation in the standards benchbook. Enter the prep information into LIMS through the "Standard Maintenance" menu; LIMS will then assign a unique S# to that standard. Write the LIMS S# and expiration date in the benchbook along with the prep information. Label the standard vial with the contents, the LIMS S#, the expiration date of the standard, and the prep chemist's initials.

Store standards at room temperature, away from light (to prevent photo-induced precipitation of silver). If the Certificate of Analysis or bottle label did not include an expiration date, assign an expiration date of one year from the date received.

Note: Prepare calibration Blanks (ICB/CCB) by adding 5.0 mL concentrated HNO_3 and 5.0 mL concentrated HCl to DI water and diluting to 100 mL.

~1 mg/L Manganese standard, used to verify lamp alignment
Dilute approximately 10µL of 10,000 mg/L Manganese standard (CPI# S4400-10M321) to 100mL in acidified (5% HCl, 5% HNO_3) deionized water. Store at room temperature for up to 1 year.

30ppm Yttrium, Internal Standard Solution
Dilute 3.0 mL of 10,000 mg/L Yttrium (Inorganic Ventures # CGY10-5) into 1L of acidified (5% HNO3, 5% HCl) deionized water. Store at room temperature for up to one year. *Note:* Because the instrument is calibrated daily, the Yttrium source standard may be used past the manufacturer's expiration date, so long as the entire sequence is analyzed using the same, freshly prepared, internal standard solution.

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Calibration Standards for the 4300DV ICP (MET-08)

	Element	CRI08 (mg/L)	CS100 (mg/L)	CS1K (mg/L)	CS10K (mg/L)	CS100K (mg/L)	Axial or Radial
Sb	Antimony	0.005	0.100	1.00	10.0	- -	A
As	Arsenic	0.005	0.100	1.00	10.0	- -	A
Ba	Barium	0.010	0.100	1.00	10.0	- -	A
Be	Beryllium	0.002	0.100	1.00	- -	- -	A
Cd	Cadmium	0.005	0.100	1.00	10.0	- -	A
Cr	Chromium	0.005	0.100	1.00	10.0	- -	A
Co	Cobalt	0.005	0.100	1.00	10.0	- -	A
Cu	Copper	0.005	0.100	1.00	10.0	- -	A
Pb	Lead	0.003	0.100	1.00	10.0	- -	A
Mo	Molybdenum	0.005	0.100	1.00	10.0	- -	A
Ni	Nickel	0.005	0.100	1.00	10.0	- -	A
Se	Selenium	0.005	0.100	1.00	10.0	- -	A
Ag	Silver	0.005	0.100	1.00	2.0	- -	A
Tl	Thallium	0.005	0.100	1.00	10.0	- -	A
V	Vanadium	0.005	0.100	1.00	10.0	- -	A
Zn	Zinc	0.005	0.100	1.00	10.0	- -	A
Al	Aluminum	0.100	- -	1.00	10.0	100.	R
Ca	Calcium	0.200	- -	1.00	10.0	100.	R
Fe	Iron	0.100	- -	1.00	10.0	100.	R
Mg	Magnesium	0.200	- -	1.00	10.0	100.	R
Mn	Manganese	0.005	0.100	1.00	10.0	- -	R
K	Potassium	0.500	- -	1.00	10.0	100.	R
Na	Sodium	0.500	- -	1.00	10.0	100.	R
B	Boron	0.100	- -	1.00	10.0	- -	A
Sn	Tin	0.040	0.100	1.00	10.0	- -	A
Ti	Titanium	0.010	0.100	1.00	10.0	- -	A

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Calibration Verification and Interference Check Standards Solutions for MET-08

Element	ICV VERT (mg/L)	CCV VERT (mg/L)	ICSA08 (mg/L)	ICSAB-T (mg/L)
Ag	5.0	5.0	- -	1.0
As	5.0	5.0	- -	0.5
Al	20.	20.	500	500
B	5.0	5.0	- -	- -
Ba	5.0	5.0	- -	0.5
Be	5.0	5.0	- -	0.5
Ca	20.	20.	500	500
Cd	5.0	5.0	- -	1.0
Co	5.0	5.0	- -	0.5
Cr	5.0	5.0	20	0.5
Cu	5.0	5.0	20	0.5
Fe	20.	20.	200	200
K	20.	20.	- -	- -
Mg	20.	20.	500	500
Mn	5.0	5.0	20	0.5
Mo	5.0	5.0	- -	0.5
Na	20.	20.	- -	- -
Ni	5.0	5.0	20	1.0
Pb	5.0	5.0	- -	1.0
Sb	5.0	5.0	- -	0.5
Se	5.0	5.0	- -	0.5
Sn	5.0	5.0	- -	- -
Ti	5.0	5.0	20	20
Tl	5.0	5.0	- -	0.5
V	5.0	5.0	20	0.5
Zn	5.0	5.0	- -	1.0

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APPENDIX_4: TARGET ELEMENTS & STANDARD REPORTING LIMITS

	Element	Water Reporting Limit (ug/L)	Soil Reporting Limit (mg/Kg)
Sb	Antimony	10.	0.50
As	Arsenic	5.0	0.25
Ba	Barium	5.0	0.25
Be	Beryllium	2.0	0.10
Cd	Cadmium	5.0	0.25
Cr	Chromium	5.0	0.25
Co	Cobalt	5.0	0.25
Cu	Copper	5.0	0.25
Pb	Lead	3.0	0.15
Mo	Molybdenum	5.0	0.25
Ni	Nickel	5.0	0.25
Se	Selenium	10.	0.50
Ag	Silver	5.0	0.25
Tl	Thallium	10.	0.50
V	Vanadium	5.0	0.25
Zn	Zinc	20.	1.0
Al	Aluminum	100	5.0
Ca	Calcium	500	25
Fe	Iron	100	5.0
Mg	Magnesium	500	25
Mn	Manganese	5.0	0.25
K	Potassium	500	25
Na	Sodium	500	25
B	Boron	100	5.0
P	Phosphorous	100	5.0
Si	Silicon	200	10.
Sn	Tin	40.	2.0
Ti	Titanium	10.	0.50

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APPENDIX_5: SPIKING LEVELS (BS, BSD, MS, MSD & SSPIKE)

Blank spike/ blank spike duplicates and sample spikes (or matrix spike/ matrix spike duplicates) should be spiked at the following levels:

	Element	Water Spiking Level (ug/L)	Soil Spiking Level (mg/Kg)
Sb	Antimony	500	25
As	Arsenic	100	5.0
Ba	Barium	2,000	100
Be	Beryllium	50	2.5
Cd	Cadmium	50	2.5.
Cr	Chromium	200	10
Co	Cobalt	500	25
Cu	Copper	250	12.5
Pb	Lead	100	5.0
Mo	Molybdenum	400	20
Ni	Nickel	500	25
Se	Selenium	100	5.0
Ag	Silver	50	2.5
Tl	Thallium	100	5.0
V	Vanadium	500	25
Zn	Zinc	500	25
Al	Aluminum	2,000	100
Ca	Calcium	20,000	1,000
Fe	Iron	1,000	50
Mg	Magnesium	20,000	1,000
Mn	Manganese	50	2.5
K	Potassium	10,000	500
Na	Sodium	20,000	1,000

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APPENDIX_6: INSTRUMENT OPERATING CONDITIONS

AXIAL / RADIAL ICP (Perkin Elmer Model 5300 DV)

Argon	Purity	99.99%
	Max Pressure	60 psi
	Min Pressure	25 psi *
Flow Rates	Plasma	17 L/min
	Auxiliary	0.4 L/min
	Nebulizer	0.55 L/min
Peristaltic Pump	Rate	1.75 mL/min
Nebulizer	Concentric Glass	Meinhard, Type A
Injector	Alumina	2.0mm
Torch	Type	Quartz
Rf Power		1450 Watts
Water Cooler	Temperature	20°C
	Pressure	30 – 60 psi
	Flow Rate	400 mL/min

**Note:* Call South Bay Welding to order an Argon delivery when the outside tank pressure is around 25-30 inches.

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APPENDIX_7: **EQUIPMENT & MAINTENANCE**

For Perkin-Elmer 5300DV ICP-AES Instruments

Any maintenance performed on the instrument must be documented in the instrument maintenance logbook. Whenever preventative or trouble-shooting maintenance is performed, document 1.) the reason the maintenance was necessary, 2.) the action taken, and 3.) the resolution of the maintenance ("passed CRI", "RSD's OK", etc.).

Perkin-Elmer's online catalog (Optima 4X00 DV heading) can be accessed at their website: http://las.perkinelmer.com/catalog/Category.aspx?CategoryName=ICP+Optical+Emission+Spectrometry+Consumables+for+Instruments or by phone at (800)-762-4000.

Tubing & Pump Windings should be replaced frequently, whenever air bubbles are present in the tubing or, for the internal standard tubing, the internal standard readings become unstable. The type of tubing can be identified by the color-coded tags at each end of the piece. Order replacement tubing from Perkin-Elmer or CPI and keep at least one spare set on-hand.

Internal Standard	Orange-Red, 0.19mm ID	P/E # N0695476
Sample Tubing	Black-Black, 2 stop	CPI # 4062-430
Drain Tubing	Red-Red, 2 stop	CPI # 4062-445

The **torch** and **injector** should be changed and cleaned whenever the torch alignment intensity drops by 20%. Immerse the parts in aqua regia over night, at minimum. Replacements can be ordered from Perkin-Elmer or CPI.

Torch:	P/E # N0770338
Copper Foil:	P/E # N0775297
Injector (2.0 mm):	P/E # N0582184

If the **nebulizer** becomes plugged, see the Operator Manual for detailed instructions.

Nebulizer (Type A):	P/E # 00472020

Lubricate the autosampler tracks approximately every six months by wiping the tracks with a Kim-Wipe saturated with 1-in-3 or clear oil.

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APPENDIX_8: USING THE WINLAB-32 SOFTWARE

Start from Overnight Standby Mode:

1.) Fill the autosampler rinsate bottle with acidified (2% HNO3, 2% HCl) DI water.
2.) Replace the pump tubing in the channel around the top of the pump then close the platen (clamp), making sure the tubing is not twisted and is centered in the clamp.
3.) Place the end of the sample uptake ("sipper") tube into a fresh aliquot of DI water.
4.) Place the end of the internal standard uptake tube into a fresh aliquot of internal standard solution.
5.) Turn on the peristaltic pump so that it rotates counterclockwise.
6.) On the computer monitor, maximize the WinLab32 window.
7.) From the main menu, select the 'File' pull-down menu, then Open, Open_Method, and click on the most recent instrument method (ex: 6010IS).
8.) Click the PLASMA icon to open the Plasma Control screen.
9.) Click ON to turn on the Plasma and automatically adjust the instrument settings to the parameters that are specified in the selected method.
10.) Allow the instrument approximately 1 hour to equilibrate.

"Profile" the Spectrometer

After the instrument has equilibrated, follow these steps to optimize the optics settings before calibration or analysis:

1.) From the main menu, select the 'Tools' pull-down menu, then Spectrometer_Control.
2.) Click 'Hg Realign' to check the axial alignment of the mercury lamp, across the diameter of the lamp.
3.) Place the sipper tube in the 1 mg/L Manganese solution.
4.) On the Spectrometer_Control window, click 'Align View'.
5.) Open the Spectra_Display to verify that the Manganese signal is getting to the detector.
6.) Verify that the 'Select Analyte' box reads [Mn 257.610]. *Note:* Manganese is used because it is a mid-range wavelength.
7.) Click ON. Instrument will then scan the response across the lamp.
8.) Check the 'Results' page to verify that lamp is properly aligned and the x-position for the maximum response is at or near 0.00.
9.) On the Spectrometer_Control window, change the Manual Settings from Axial to Radial.
10.) Click 'Align View'.
11.) Click 'ON'.
12.) From the main menu, select File, Print, Active_Window.
13.) Close 'Results'.
14.) Close 'Spectrometer Control'.
15.) Write the positions & intensities in the instrument maintenance log.
16.) Move the sipper tube to the rinse DI and allow the system to rinse for about 1 minute while you start writing the sequence.

Set Up a Sequence

1.) In the online session, open METHOD_EDITOR.

a.) Open the method you want to use.
b.) Verify that the LIMS S# is correct for each standard.
c.) Close the Method Editor.

2.) Click the SamInfo icon and enter the sample information under the 'Parameters that Vary by Sample' heading.
   a.) For samples (or batch QC), the first A/S Location will be 11, because spots 1 through 10 are always used for the standards.
   b.) The standards are not entered into the sequence because the identities and frequencies are specified in the software method.
   c.) In the 'Sample ID' column, enter sample information in the format:
      Sample #, batch#, IDF
      MB, QC#, batch#, IDF
      LCS, QC#, batch#, IDF
      Etc…
         Where:
         Samplenum is the LIMS sample number (ie: 160961-005)
         QC# is the LIMS ID of the batch QC sample (ie: QC16935)
         Batch# is the LIMS batch number (ie: 95687)
         IDF is the instrument dilution factor for the sample, written as "5" or similar
   d.) To insert instrument blanks (rinses), set the A/S Location to '0' (zero) and the sample ID to 'rinse' or IB.
   e.) When your sequence is complete, pull-down the File menu, and select Save_As, Sample Info File, and name the file with the day's date (ex: 092404.sif).
      Important! Make sure to save the Sample Info File as an SIF, *not* as the method!
   f.) From the File pull-down menu, select Print, Active_Window to print the sequence.
   g.) Close the Sample Info File window.

3.) Load the autosampler.
4.) Verify the sample locations in the autosampler tray against the sequence.
5.) Start the sequence.
   a.) From the main menu, click the AUTO icon (to use the autosampler locations).
   b.) In the SetUp sheet, call up the most recent Sample Info File.
   c.) "Results Data Set Name".
   d.) Type in the date as MMDDYY (ex: 092404).
   e.) Make sure the following are checked for use:
      Use Sample Info — Save Data
      Print Log during analysis — <don't check "Auto export">
      Auto Wavelength Realign (every 60 seconds)
   f.) Verify that the correct method and sample info file have been called up.
   g.) Choose which type of run you need to start:
      CALIBRATE - will only run the beginning calibration standards
      ANALYZE ALL - will run the whole sequence
      ANALYZE SAMPLES - runs only the samples after the ICAL standards
   h.) Click OK.

6.) Leave the window open so you can verify the status of the sequence as it is running. This window can be minimized if you are working in another window, but don't close it or the instrument will immediately stop running the sequence!

Send Data to LIMS

1.) Click the DATA MANAGER icon on the desktop.
2.) Enter the current sequence name into the 'Result Name' field.
3.) Click the EXPORT button at the top of the screen.
4.) Select 'Create', then 'Browse'.
5.) Find the LIMS.xpt file and 'Open' the file.
6.) Click the FINISH button at the bottom of the screen.
7.) Then EXPORT and FINISH sending the data to LIMS.

Edit a Sequence

If you need to add samples to the sequence, you must stop the instrument run and edit the sequence through the SamInfo screen, *then* go to the 'Analyze' sheet and 'Rebuild List'. Print the active window to print the modified sequence.

Use the Priority button on the 'Analyze' sheet to do immediate reanalysis of the sample. The software will automatically reposition the CCV & CCB, as the methods specify the frequency of their analysis.

Reprocess Data

Use the offline session to reprocess old data files, when needed.

1.) Maximize the 'WinLab32 Offline' window.
2.) Verify that the method named in the upper right corner, next to the "Method' label, is the method that you want to use. If not, open the correct method.
3.) Click REPROC.
4.) Select the Data Set to be reprocessed and save it with an '-R' suffix.

View/Print Uncorrected Intensities

If a client, validator or auditor asks for the original, uncorrected (for internal standard) intensities, use the following steps to view or print those readings:

1.) Maximize the 'WinLab32 Offline' window.
2.) Select the sequence you need to reprocess.
3.) Verify that the method named in the upper right corner, next to the "Method' label, is the method that you want to use. If not, open the correct method.
4.) Go to the menu bar and select Process > Internal Standard.
5.) Remove the IS assignment from each element. *Caution:* Don't save this version unless you save it with a different method name, or else it will overwrite the original data when you reprocess it and you will have to go back and redo it again.
6.) Go to the sequence and highlight the calibration and data files you want to reprocess.
7.) Click REPROC to reprocess those files and either view the readings onscreen or send them to a local printer.

APPENDIX_9: DATA MANAGEMENT

All of the following database functions are accessed from within the Database Manager Utility. Both the online and offline versions of Optima WinLab32 should be closed while performing these functions.

1. **Check the Library** (should be done weekly)
    a. Open the **Data Manager Utility.**
    b. Click on the **Check** button to run a check of the **Results** database.
    c. Change the Library Category from Results to **Methods.**
    d. Click on the **Check** button to run a check of the Methods database.

2. **Archive Files in the Results Library** (should be done bimonthly, in the second week of the month following the two month period to be archived. For example, March and April should be archived in the week of May 15th.)
    a. Change **back to the Results library** if needed.
    b. Selecting one month's worth at a time, **highlight the files you want to archive**. You can either click and shift click to highlight all files in between the two selected files, or click and Ctrl click to select individual files.
    c. **Select the Archive box.** Select OK.
    d. The files that are to be archived will appear in the window. Use the **browse button** to **select the location** for the **archived file**. Use the defaults location (C:\pe\Curtis and Tompkins\Archived Data\).
    e. The archive name is automatically selected. The file name will start with MS for a methods archive, and RS for a results archive. The rest of the file name will be the six characters representing the date the archive was created. **<u>CHANGE</u> THE DATE TO REPRESENT THE DATE OF THE FILES ARCHIVED IN THE FORMAT RSMMYYYY.ZIP.**
    f. Select OK and the archive will be created.
    g. Repeat a –f above, changing the location for the archived file to G:\met08\.

3. **Deleting Archived Files From the Results Database**
    h. After the month's archives have been created on both the C: drive and on the G: drive, **highlight the files** you want deleted.
    i. Click on the **delete box**.
    j. The selected files that are to be deleted will be listed. Make sure you are deleting the correct files!
    k. Select OK, the files are now deleted.

4. **Packing The Results Library** (should be done after deleting files from the Database)
    l. **Click** on the **Pack button.**

APPENDIX_10: INTERFERENCES

Matrix interferences may cause inaccuracies in the determined element concentrations. These interferences generally fall into three categories: spectral interferences, chemical interferences, and physical interferences.

Spectral interferences may include:

1.) The overlap of the spectral line of one element with another, which can be minimized through use of interelement correction (IEC) factors. The IEC factors are determined annually. High levels of aluminum, calcium, iron, and magnesium are often responsible for this type of interference. The daily ICSAB standard is used verify the correction factors for contributions from high levels of these elements.

2.) Unresolved overlap of molecular spectra, which may require selection of an alternate wavelength for measurement or dilution and reanalysis of the sample.

3.) Background contributions from continuous or recombination phenomena, or from the emissions of elements present at high concentrations. Background correction can compensate for these effects by measuring the emissions adjacent to the analyte line.

Chemical interferences are characterized by molecular compound formation, ionization effects, and vaporization effects. These types of interferences are highly matrix dependent and are not often seen in the ICP analysis, since the plasma will dissociate most compounds.

Physical interferences are typically those occurring during the digestion, nebulization, and transport. Common physical interferences include:

1.) Loss of volatile elements (antimony) during the digestion process if the digestate is superheated or allowed to go dry during heating.

2.) Precipitation of certain elements (silver) during the digestion process if present in relatively high levels.

3.) Zinc contamination due to dusty surroundings.

4.) Differences in sample viscosity and surface tension due to high levels of dissolved solids. Serial dilutions may be used to identify this type of interference, which may clog the nebulizer and tubing.

This SOP contains information that may not be disseminated to entities other than C&T staff, clients, and regulators.

APPENDIX_11: **EPA 200.7**

Metals in Drinking Water & Wastewater

The following criteria and discussion apply to any samples submitted for the EPA 200.7 analysis:

INITIAL CALIBRATION:
Method 200.7 only requires a single-point calibration and does not specify a correlation coefficient (r) criteria but C&T analyzes a multi-point calibration with an acceptance criterion of r $\geq$0.995. The same general calibration criteria must be followed as for 6010 (ie: intermediate levels may not be dropped, the low point must be at or below the reporting limit, etc.).

CALIBRATION VERIFICATION (ICV/CCV):
Method 200.7 requires that ICV and CCV concentrations be obtained by using the average of replicate exposures, with an RSD of $\leq$ 3% between exposures. The ICV and CCV recoveries must be within 5% of the true concentration (95-105% recovery).

INTERFERENCE CHECK STANDARDS (ICS-A and ICS-AB):
ICS-AB standards containing both common interferents and low-level target analytes should be analyzed at the beginning of each sequence, after the calibration standards and verifications, and at the end of the sequence. The recovery for each analyte should be within 80-120% of the true concentration or the associated sample results for that element may not be reported.

EPA 200.7 does not mention the ICS-A standard; C&T standard practice is to include this standard in the analytical sequence, following SW-846 guidance as described in the main body of this SOP. The determined concentration of the non-interferent should be no more than $\pm$RL in either direction.

SAMPLE ANALYSIS:
EPA 200.7 requires that the samples be aspirated for at least 30 seconds prior to collection of data to allow the response to stabilize; C&T aspirates the samples for approximately 30 seconds or longer when necessary, as described in the main body of this document.

EPA 200.7 requires a minimum of 3 integrations for each mass, reporting the average of the three; this matches C&T standard practice as described in the main body of this SOP. The method does not discuss a limit on the RSD between these integrations; C&T has established an internal requirement of 20%RSD between the integrations. If the RSD is >20, the sample must be rerun and/or diluted to reduce matrix interferences.

BATCH QC:

- Method Blank: EPA 200.7 requires redigestion and reanalysis of any sample associated with a method blank containing laboratory contamination greater than **2.2x the MDL** for that element and <10x the concentration in the sample. If the sample is ND for the blank

contaminant or is >10x the level found in the blank, the sample may be reported without corrective action.

- BS/BSD: EPA 200.7 specifies LCS (or LFB, "Lab Fortified Blank") recovery limits of **85-115%** but does not discuss the use of a duplicate. C&T digests and analyzes a pair of blank spikes with every batch to better monitor both the accuracy and precision of the process; RPD limits are based on statistically generated control limits.

- MS/MSD: EPA 200.7 specifies recovery limits of **70-130%** but does not discuss the use of a duplicate. C&T digests and analyzes a pair of matrix spikes with every batch to better monitor both the accuracy and precision of the process on real-world samples; RPD limits are based on statistically generated control limits.

- Serial Dilution recovery limits are 95-105%.

- Post Digestion Spike recovery limits are 90-110%

INITIAL DEMONSTRATION OF CAPABILITY
Each new analyst must complete an "IDOC" consisting of 4 consecutive LCS/BS/BSD batch QC samples that pass recovery and RPD/RSD limits. For EPA 200.7, the IDOC also includes analysis of an MDL study.

INSTRUMENT DETECTION LIMIT (IDL) STUDY
IDL studies must be completed quarterly by analyzing a reagent blank for seven consecutive injections on each of 3 non-consecutive days. The RSD is determined for each element on each day then the 3 RSD's are averaged to determine the IDL.

This SOP contains information that may not be disseminated to entities other than C&T staff, clients, and regulators.

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Attachment 4: Control Limits

EPA 608, EPA 8082, EPA 8082A QC Limits

C&T In-House Limits for
Feb 11, 2013 - Aug 10, 2013

Matrix	Analyte	LCS/LCSD Recovery	LCS/LCSD RPD	MS/MSD Recovery	MS/MSD RPD
Water	Aroclor-1016	69 - 131	31	46 - 155	51
	Aroclor-1221	60 - 140	20	50 - 150	30
	Aroclor-1232	60 - 140	20	50 - 150	30
	Aroclor-1242	80 - 176	20	70 - 186	30
	Aroclor-1248	78 - 165	20	68 - 175	30
	Aroclor-1254	60 - 140	20	50 - 150	30
	Aroclor-1260	56 - 130	40	28 - 141	52
	Aroclor-1262	60 - 140	20	50 - 150	30
	Aroclor-1268	60 - 140	20	50 - 150	30
	TCMX	47 - 120		47 - 120	
	Decachlorobiphenyl	33 - 120		33 - 120	

C&T internal list numbers: 83990

EPA 608, EPA 8082, EPA 8082A QC Limits

C&T In-House Limits for
Feb 11, 2013 - Aug 10, 2013

Matrix	Analyte	LCS/LCSD Recovery	LCS/LCSD RPD	MS/MSD Recovery	MS/MSD RPD
Soil	Aroclor-1016	64 - 143	39	58 - 155	44
	Aroclor-1221	60 - 140	20	50 - 150	30
	Aroclor-1232	60 - 140	20	50 - 150	30
	Aroclor-1242	60 - 140	20	50 - 150	30
	Aroclor-1248	60 - 140	20	50 - 150	30
	Aroclor-1254	44 - 169	20	34 - 179	30
	Aroclor-1260	58 - 146	54	35 - 159	53
	Aroclor-1262	60 - 140	20	50 - 150	30
	Aroclor-1268	60 - 140	20	50 - 150	30
	TCMX	66 - 142		66 - 142	
	Decachlorobiphenyl	43 - 139		43 - 139	

C&T internal list numbers: 83992

EPA 8015B, EPA 8015D, SW8015AZR1 QC Limits

C&T In-House Limits for
Feb 11, 2013 - Aug 10, 2013

Matrix	Analyte	LCS/LCSD Recovery	LCS/LCSD RPD	MS/MSD Recovery	MS/MSD RPD
Water	JP-8, C10-C16	50 - 150	20	40 - 150	30
	JP-5 C10-C16	60 - 140	20	50 - 150	30
	Jet Fuel A C10-C16	60 - 140	20	22 - 120	30
	Kerosene C10-C16	50 - 150	20	40 - 150	30
	Diesel C10-C14	50 - 120	45	50 - 120	55
	Diesel C10-C20	58 - 120	46	59 - 120	45
	Diesel C10-C22	58 - 120	47	60 - 120	44
	Diesel C10-C24	59 - 120	46	61 - 120	43
	Diesel C10-C28	59 - 120	46	60 - 120	41
	Diesel C10-C40	53 - 129	42	43 - 139	52
	Diesel C12-C22	61 - 120	48	64 - 120	51
	Diesel C12-C24	62 - 120	47	64 - 120	49
	Diesel C12-C28	63 - 120	46	62 - 120	27
	Diesel C12-C32	61 - 120	45	62 - 120	48
	Diesel C12-C36	60 - 140	20	50 - 150	30
	Diesel C14-C28	71 - 121	48	68 - 124	57
	Pro-Par Clearant C10-C16	60 - 140	20	50 - 150	30
	Diesel C10-C12	60 - 140	20	50 - 150	30
	Diesel C12-C16	60 - 140	20	50 - 150	30
	Diesel C16-C24	53 - 124	49	49 - 124	52
	Diesel C12-C14	60 - 140	20	50 - 150	30
	Free Product C10-C24	50 - 150	20	40 - 150	30
	Diesel C14-C16	60 - 140	20	50 - 150	30
	Diesel C16-C18	60 - 140	20	50 - 150	30
	Trimsol C12-C36	50 - 150	20	40 - 150	30
	Diesel C18-C20	60 - 140	20	50 - 150	30
	Diesel C20-C22	60 - 140	20	50 - 150	30
	Diesel C22-C24	60 - 140	20	50 - 150	30
	Motor Oil C20-C36	60 - 140	20	50 - 150	30
	Motor Oil C20-C40	50 - 150	25	50 - 150	25
	Motor Oil C22-C32	60 - 140	20	50 - 150	30
	Motor Oil C22-C36	60 - 140	20	50 - 150	30
	Motor Oil C24-C36	60 - 140	20	50 - 150	30
	Motor Oil C28-C36	60 - 140	20	50 - 150	30
	Motor Oil C28-C40	60 - 140	20	50 - 150	30
	Motor Oil C20-C50	52 - 150	20	42 - 160	30
	Hydraulic Fluid, C12-40	60 - 140	20	50 - 150	30
	Ash Creek Crude C10-C40	50 - 150	25	50 - 150	25
	Crude Oil C10-C40	50 - 150	25	50 - 150	25
	Hydraulic Fluid, C24-36	60 - 140	20	50 - 150	30
	Creosote C10-C36	50 - 150	25	50 - 150	25
	Bunker C C12-40	60 - 140	20	50 - 150	30
	Bunker C C12-50	60 - 140	20	50 - 150	30
	C12-C40 as P86-9/10	60 - 140	20	50 - 150	30
	C12-C40 as MW11-54	60 - 140	20	50 - 150	30
	Transformer Oil C12-C40	50 - 150	25	50 - 150	25
	Transformer Oil C12-C50	50 - 150	25	50 - 150	25
	Mineral Oil C12-C40	50 - 150	25	50 - 150	25

EPA 8015B, EPA 8015D, SW8015AZR1 QC Limits

C&T In-House Limits for
Feb 11, 2013 - Aug 10, 2013

Matrix	Analyte	LCS/LCSD Recovery	LCS/LCSD RPD	MS/MSD Recovery	MS/MSD RPD
	Tetrabutyl orthosilicate	50 - 150	25	50 - 150	25
	Fuel Oil C24-C36	50 - 150	25	50 - 150	25
	o-Terphenyl	62 - 133		62 - 133	
	Hexacosane	69 - 144		69 - 144	

C&T internal list numbers: 83975

EPA 200.8, EPA 6020, EPA 6020A QC Limits

C&T In-House Limits
effective: Feb 11, 2013

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
Water	Aluminum	71 - 122	23	66 - 126	20
	Antimony	72 - 120	20	66 - 120	26
	Arsenic	76 - 120	20	68 - 122	33
	Barium	75 - 120	25	65 - 123	30
	Beryllium	61 - 120	25	63 - 125	28
	Cadmium	74 - 120	25	69 - 120	31
	Calcium	70 - 120	24	51 - 129	28
	Chromium	75 - 120	21	68 - 120	25
	Cobalt	76 - 120	20	69 - 120	20
	Copper	67 - 123	22	59 - 123	34
	Iron	69 - 125	31	62 - 128	39
	Lead	76 - 120	22	70 - 120	21
	Magnesium	67 - 123	22	54 - 132	36
	Manganese	77 - 120	20	66 - 120	25
	Molybdenum	72 - 120	21	69 - 120	30
	Nickel	72 - 120	23	62 - 120	37
	Potassium	69 - 128	29	54 - 132	45
	Selenium	75 - 120	32	63 - 124	34
	Silver	80 - 120	22	70 - 120	22
	Sodium	59 - 132	28	37 - 149	42
	Thallium	76 - 120	22	72 - 120	21
	Thorium	80 - 120	20	45 - 139	20
	Uranium	80 - 120	20	69 - 128	20
	Vanadium	74 - 120	20	67 - 120	39
	Zinc	71 - 123	32	57 - 130	38
Soil	Aluminum	59 - 166	46	54 - 145	60
	Antimony	80 - 120	20	19 - 120	32
	Arsenic	80 - 120	20	73 - 120	32
	Barium	80 - 120	20	49 - 142	33
	Beryllium	61 - 124	29	65 - 122	37
	Cadmium	80 - 120	20	74 - 120	39
	Calcium	75 - 120	20	56 - 134	23
	Chromium	80 - 120	20	58 - 131	28
	Cobalt	80 - 120	20	70 - 122	25
	Copper	80 - 126	20	65 - 128	66
	Iron	76 - 127	33	51 - 144	35
	Lead	80 - 120	20	68 - 127	34
	Magnesium	72 - 132	20	53 - 139	43
	Manganese	80 - 120	20	56 - 140	20
	Molybdenum	80 - 120	20	69 - 120	30
	Nickel	79 - 122	20	59 - 136	42
	Potassium	76 - 130	20	58 - 135	49
	Selenium	76 - 126	30	65 - 122	33
	Silver	80 - 126	20	80 - 125	34
	Sodium	70 - 139	30	59 - 129	23
	Thallium	79 - 120	20	75 - 120	35

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
	Thorium	80 - 120	20	70 - 130	30
	Titanium	80 - 120	20	70 - 130	30
	Uranium	80 - 120	20	70 - 130	30
	Vanadium	80 - 120	20	55 - 134	23
	Zinc	79 - 128	27	49 - 145	51

C&T internal list numbers: 84024, 84026

EPA 200.7, EPA 6010B, EPA 6010C QC Limits

C&T In-House Limits
effective: Feb 11, 2013

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
Water	Aluminum	70 - 120	21	61 - 125	21
	Antimony	75 - 120	20	74 - 120	20
	Arsenic	78 - 120	22	74 - 130	23
	Barium	80 - 120	20	75 - 120	23
	Beryllium	80 - 120	20	80 - 123	20
	Cadmium	80 - 120	20	72 - 121	20
	Calcium	80 - 120	20	62 - 130	22
	Chromium	80 - 120	20	74 - 120	20
	Cobalt	79 - 120	20	73 - 120	20
	Copper	77 - 120	20	73 - 121	21
	Iron	77 - 120	24	64 - 129	22
	Lead	78 - 120	20	68 - 120	24
	Magnesium	79 - 120	20	68 - 121	20
	Manganese	80 - 120	20	67 - 130	24
	Molybdenum	80 - 120	20	78 - 120	20
	Nickel	80 - 120	20	73 - 120	20
	Potassium	74 - 120	20	67 - 130	22
	Selenium	75 - 120	25	67 - 129	39
	Silver	77 - 120	20	62 - 124	20
	Sodium	76 - 121	20	58 - 136	30
	Thallium	79 - 120	23	67 - 120	24
	Vanadium	80 - 120	20	80 - 120	20
	Zinc	80 - 120	20	72 - 123	20
	Boron	80 - 120	20	73 - 125	20
	Phosphorus	80 - 120	20	70 - 130	30
	Silicon	80 - 120	20	70 - 130	30
	Sulfide	80 - 120	20	70 - 130	30
	Tin	78 - 120	20	73 - 120	20
	Strontium	80 - 120	20	70 - 130	30
	Titanium	78 - 120	20	75 - 125	20
Soil	Aluminum	80 - 120	20	43 - 167	49
	Antimony	80 - 120	20	8 - 120	29
	Arsenic	80 - 120	20	71 - 121	34
	Barium	80 - 120	20	48 - 133	45
	Beryllium	80 - 120	20	78 - 120	20
	Cadmium	80 - 120	20	69 - 120	23
	Calcium	80 - 120	20	38 - 154	47
	Chromium	80 - 120	20	60 - 122	34
	Cobalt	80 - 120	20	61 - 120	37
	Copper	80 - 120	20	44 - 151	35
	Iron	80 - 120	20	42 - 146	51
	Lead	80 - 120	22	52 - 120	51
	Magnesium	80 - 120	20	34 - 154	39
	Manganese	80 - 120	20	43 - 151	53
	Molybdenum	80 - 120	20	67 - 120	20
	Nickel	80 - 120	20	45 - 134	38

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
	Potassium	80 - 120	20	50 - 147	32
	Selenium	80 - 120	20	67 - 120	27
	Silver	80 - 120	20	66 - 120	30
	Sodium	80 - 121	20	64 - 134	32
	Thallium	80 - 120	20	62 - 120	20
	Vanadium	80 - 120	20	55 - 137	30
	Zinc	80 - 120	20	38 - 146	36
	Boron	80 - 120	20	52 - 120	33
	Phosphorus	80 - 120	20	70 - 130	30
	Silicon	80 - 120	20	70 - 130	30
	Sulfide	80 - 120	20	70 - 130	30
	Strontium	80 - 120	20	70 - 130	30
	Tin	80 - 120	20	63 - 120	27
	Titanium	80 - 120	20	20 - 163	67

C&T internal list numbers: 84020, 84022

EPA 245.1, EPA 7470A, EPA 7471B QC Limits

C&T In-House Limits
effective: Feb 11, 2013

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
Water	Mercury	80 - 120	20	62 - 124	35
Soil	Mercury	80 - 120	20	72 - 135	42

C&T internal list numbers: 84016, 84018

EPA 8015B, EPA 8015D, SW8015AZR1 QC Limits

C&T In-House Limits
effective: Feb 11, 2013

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
Water	JP-8, C10-C16	50 - 150	20	40 - 150	30
	JP-5 C10-C16	60 - 140	20	50 - 150	30
	Jet Fuel A C10-C16	60 - 140	20	22 - 120	30
	Kerosene C10-C16	50 - 150	20	40 - 150	30
	Diesel C10-C14	50 - 120	45	50 - 120	55
	Diesel C10-C20	58 - 120	46	59 - 120	45
	Diesel C10-C22	58 - 120	47	60 - 120	44
	Diesel C10-C24	59 - 120	46	61 - 120	43
	Diesel C10-C28	59 - 120	46	60 - 120	41
	Diesel C10-C40	53 - 129	42	43 - 139	52
	Diesel C12-C22	61 - 120	48	64 - 120	51
	Diesel C12-C24	62 - 120	47	64 - 120	49
	Diesel C12-C28	63 - 120	46	62 - 120	27
	Diesel C12-C32	61 - 120	45	62 - 120	48
	Diesel C12-C36	60 - 140	20	50 - 150	30
	Diesel C14-C28	71 - 121	48	68 - 124	57
	Pro-Par Clearant C10-C16	60 - 140	20	50 - 150	30
	Diesel C10-C12	60 - 140	20	50 - 150	30
	Diesel C12-C16	60 - 140	20	50 - 150	30
	Diesel C16-C24	53 - 124	49	49 - 124	52
	Diesel C12-C14	60 - 140	20	50 - 150	30
	Free Product C10-C24	50 - 150	20	40 - 150	30
	Diesel C14-C16	60 - 140	20	50 - 150	30
	Diesel C16-C18	60 - 140	20	50 - 150	30
	Trimsol C12-C36	50 - 150	20	40 - 150	30
	Diesel C18-C20	60 - 140	20	50 - 150	30
	Diesel C20-C22	60 - 140	20	50 - 150	30
	Diesel C22-C24	60 - 140	20	50 - 150	30
	Motor Oil C20-C36	60 - 140	20	50 - 150	30
	Motor Oil C20-C40	50 - 150	25	50 - 150	25
	Motor Oil C22-C32	60 - 140	20	50 - 150	30
	Motor Oil C22-C36	60 - 140	20	50 - 150	30
	Motor Oil C24-C36	60 - 140	20	50 - 150	30
	Motor Oil C28-C36	60 - 140	20	50 - 150	30
	Motor Oil C28-C40	60 - 140	20	50 - 150	30
	Motor Oil C20-C50	52 - 150	20	42 - 160	30
	Hydraulic Fluid, C12-40	60 - 140	20	50 - 150	30
	Ash Creek Crude C10-C40	50 - 150	25	50 - 150	25
	Crude Oil C10-C40	50 - 150	25	50 - 150	25
	Hydraulic Fluid, C24-36	60 - 140	20	50 - 150	30
	Creosote C10-C36	50 - 150	25	50 - 150	25
	Bunker C C12-40	60 - 140	20	50 - 150	30
	Bunker C C12-50	60 - 140	20	50 - 150	30
	C12-C40 as P86-9/10	60 - 140	20	50 - 150	30
	C12-C40 as MW11-54	60 - 140	20	50 - 150	30
	Transformer Oil C12-C40	50 - 150	25	50 - 150	25
	Transformer Oil C12-C50	50 - 150	25	50 - 150	25

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
	Mineral Oil C12-C40	50 - 150	25	50 - 150	25
	Tetrabutyl orthosilicate	50 - 150	25	50 - 150	25
	Fuel Oil C24-C36	50 - 150	25	50 - 150	25
	o-Terphenyl	62 - 133		62 - 133	
	Hexacosane	69 - 144		69 - 144	
Soil	JP-5 C10-C16	60 - 140	20	80 - 127	30
	JP-8, C10-C16	50 - 150	20	40 - 150	30
	Diesel C10-C14	51 - 143	20	45 - 139	46
	Jet Fuel A C10-C16	50 - 150	20	40 - 150	30
	Diesel C14-C28	63 - 136	20	35 - 155	48
	Kerosene C10-C16	60 - 140	20	50 - 150	30
	Diesel C10-C20	62 - 129	20	46 - 136	45
	Diesel C10-C22	62 - 130	20	42 - 143	45
	Diesel C10-C24	62 - 130	20	39 - 148	45
	Diesel C10-C28	62 - 130	20	30 - 158	49
	Diesel C10-C40	61 - 130	20	51 - 140	30
	Diesel C12-C22	64 - 130	20	42 - 148	42
	Diesel C12-C24	63 - 130	20	38 - 152	43
	Diesel C12-C28	62 - 127	20	27 - 163	48
	Diesel C12-C32	64 - 130	20	25 - 162	53
	Diesel C12-C36	60 - 140	20	50 - 150	30
	Diesel C10-C12	60 - 140	20	50 - 150	30
	Diesel C12-C16	60 - 140	20	50 - 150	30
	Diesel C16-C24	62 - 132	20	31 - 162	46
	Trimsol C12-C36	50 - 150	25	50 - 150	25
	Motor Oil C20-C36	64 - 140	20	54 - 150	30
	Motor Oil C20-C40	50 - 150	35	50 - 150	35
	Motor Oil C22-C32	63 - 142	20	53 - 152	30
	Motor Oil C22-C36	63 - 139	20	53 - 149	30
	Motor Oil C24-C36	61 - 140	20	51 - 150	30
	Motor Oil C24-C50	50 - 150	20	40 - 150	30
	Motor Oil C28-C36	57 - 140	20	47 - 150	30
	Motor Oil C28-C40	55 - 142	20	45 - 152	30
	Motor Oil C20-C50	50 - 150	35	50 - 150	35
	Hydraulic Fluid, C10-40	60 - 140	20	50 - 150	30
	Hydraulic Fluid, C12-40	60 - 140	20	50 - 150	30
	Ash Creek Crude C10-C40	50 - 150	20	40 - 150	30
	Crude Oil C10-C40	50 - 150	20	40 - 150	30
	Hydraulic Fluid, C24-36	50 - 150	20	40 - 150	30
	Creosote C10-C36	50 - 150	20	40 - 150	30
	Bunker C C10-C40	35 - 127	20	1 - 161	77
	Bunker C C12-40	60 - 140	20	50 - 150	30
	Bunker C C12-50	34 - 122	20	7 - 143	57
	TPH-b(PM) C10-C40	35 - 127	20	1 - 161	77
	C12-C40 as P86-9/10	35 - 127	20	1 - 161	77
	TPH-d+b(PM) C10-C40	35 - 127	20	1 - 161	77
	C12-C40 as MW11-54	35 - 127	20	1 - 161	77
	Transformer Oil C12-C40	50 - 150	35	50 - 150	35
	Transformer Oil C12-C50	50 - 150	35	50 - 150	35
	Mineral Oil C12-C40	50 - 150	35	50 - 150	35
	Tetrabutyl orthosilicate	50 - 150	35	50 - 150	35
	Fuel Oil C24-C36	50 - 150	35	50 - 150	35
	TPH (C12-C36)	50 - 150	35	50 - 150	35
	Diesel C12-C14	60 - 140	20	50 - 150	30

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
	Diesel C14-C16	60 - 140	20	50 - 150	30
	Diesel C16-C18	60 - 140	20	50 - 150	30
	Diesel C18-C20	60 - 140	20	50 - 150	30
	Diesel C20-C22	60 - 140	20	50 - 150	30
	Diesel C22-C24	60 - 140	20	50 - 150	30
	o-Terphenyl	62 - 136		62 - 136	
	Hexacosane	55 - 151		55 - 151	

C&T internal list numbers: 83975, 83976

EPA 608, EPA 8081A, EPA 8081B QC Limits

C&T In-House Limits
effective: Feb 11, 2013

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
Water	gamma-BHC	68 - 128	27	49 - 164	46
	Heptachlor	59 - 121	20	65 - 140	37
	Aldrin	62 - 127	20	68 - 135	40
	Dieldrin	66 - 139	28	52 - 165	36
	Endrin	55 - 144	31	46 - 172	39
	4,4'-DDT	48 - 144	30	43 - 173	29
	Isodrin	61 - 127	24	51 - 144	36
	alpha-BHC	69 - 130	20	64 - 147	43
	beta-BHC	67 - 136	31	64 - 154	64
	delta-BHC	65 - 138	20	55 - 172	27
	Heptachlor epoxide	64 - 130	24	56 - 147	39
	Heptachlor epoxide B	68 - 120	37	58 - 130	47
	gamma-Chlordane	62 - 132	24	59 - 141	28
	alpha-Chlordane	63 - 128	34	61 - 141	33
	Endosulfan I	65 - 130	30	43 - 159	42
	4,4'-DDE	60 - 139	32	54 - 156	23
	Endosulfan II	64 - 141	26	44 - 157	36
	Endosulfan sulfate	49 - 149	28	28 - 180	29
	4,4'-DDD	54 - 146	31	62 - 164	22
	Endrin aldehyde	60 - 147	23	38 - 159	63
	Chlordane (Technical)	60 - 140	20	50 - 150	30
	Methoxychlor	37 - 152	35	28 - 200	57
	Endrin ketone	52 - 179	29	64 - 195	33
	Toxaphene	60 - 140	20	50 - 150	30
	TCMX	38 - 133		38 - 133	
	Decachlorobiphenyl	31 - 123		31 - 123	
Soil	gamma-BHC	49 - 120	20	47 - 130	38
	Heptachlor	44 - 121	20	39 - 137	47
	Aldrin	53 - 120	20	44 - 140	44
	Dieldrin	42 - 141	20	45 - 146	37
	Endrin	45 - 137	20	45 - 146	42
	4,4'-DDT	37 - 142	20	30 - 151	47
	Isodrin	44 - 124	20	44 - 135	36
	alpha-BHC	50 - 121	20	50 - 129	37
	beta-BHC	54 - 124	20	48 - 135	39
	delta-BHC	48 - 133	20	52 - 137	48
	Heptachlor epoxide	52 - 122	20	46 - 138	40
	Heptachlor epoxide B	37 - 129	20	39 - 130	58
	gamma-Chlordane	52 - 125	20	41 - 147	40
	alpha-Chlordane	52 - 124	20	42 - 142	45
	Endosulfan I	50 - 126	20	49 - 138	40
	4,4'-DDE	51 - 133	20	44 - 148	37
	Endosulfan II	42 - 137	20	38 - 137	48
	Endosulfan sulfate	36 - 134	20	38 - 141	52
	4,4'-DDD	47 - 139	20	45 - 149	40

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
	Endrin aldehyde	15 - 123	20	27 - 125	46
	Endrin ketone	40 - 164	20	33 - 166	49
	Chlordane (Technical)	60 - 140	20	50 - 150	30
	Methoxychlor	37 - 144	20	40 - 147	44
	Toxaphene	60 - 140	20	50 - 150	30
	TCMX	45 - 129		45 - 129	
	Decachlorobiphenyl	32 - 132		32 - 132	

C&T internal list numbers: 83984, 83988

EPA 608, EPA 8082, EPA 8082A QC Limits

C&T In-House Limits
effective: Feb 11, 2013

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
Water	Aroclor-1016	69 - 131	31	46 - 155	51
	Aroclor-1221	60 - 140	20	50 - 150	30
	Aroclor-1232	60 - 140	20	50 - 150	30
	Aroclor-1242	80 - 176	20	70 - 186	30
	Aroclor-1248	78 - 165	20	68 - 175	30
	Aroclor-1254	60 - 140	20	50 - 150	30
	Aroclor-1260	56 - 130	40	28 - 141	52
	Aroclor-1262	60 - 140	20	50 - 150	30
	Aroclor-1268	60 - 140	20	50 - 150	30
	TCMX	47 - 120		47 - 120	
	Decachlorobiphenyl	33 - 120		33 - 120	
Soil	Aroclor-1016	64 - 143	39	58 - 155	44
	Aroclor-1221	60 - 140	20	50 - 150	30
	Aroclor-1232	60 - 140	20	50 - 150	30
	Aroclor-1242	60 - 140	20	50 - 150	30
	Aroclor-1248	60 - 140	20	50 - 150	30
	Aroclor-1254	44 - 169	20	34 - 179	30
	Aroclor-1260	58 - 146	54	35 - 159	53
	Aroclor-1262	60 - 140	20	50 - 150	30
	Aroclor-1268	60 - 140	20	50 - 150	30
	TCMX	66 - 142		66 - 142	
	Decachlorobiphenyl	43 - 139		43 - 139	

C&T internal list numbers: 83990, 83992

EPA 8270C-SIM, EPA 8270D-SIM QC Limits

C&T In-House Limits
effective: Feb 11, 2013

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
Water	1,4-Dioxane	41 - 120	45	20 - 129	44
	Pentachlorophenol	60 - 140	20	50 - 150	30
	Naphthalene	63 - 120	39	65 - 120	26
	1-Methylnaphthalene	62 - 120	35	58 - 120	53
	2-Methylnaphthalene	62 - 120	39	56 - 120	54
	Acenaphthylene	50 - 120	41	50 - 120	26
	Acenaphthene	52 - 120	72	49 - 120	32
	Fluorene	54 - 120	39	49 - 120	26
	Phenanthrene	48 - 120	35	50 - 120	26
	Anthracene	44 - 120	47	40 - 120	30
	Fluoranthene	45 - 120	37	48 - 120	25
	Pyrene	45 - 120	53	41 - 121	26
	Benzo(a)anthracene	41 - 120	40	47 - 120	23
	Chrysene	44 - 120	37	46 - 120	35
	Benzo(b)fluoranthene	41 - 120	47	48 - 120	52
	Benzo(k)fluoranthene	39 - 120	46	43 - 120	44
	Benzo(a)pyrene	40 - 120	53	48 - 120	38
	Indeno(1,2,3-cd)pyrene	33 - 120	51	34 - 120	47
	Dibenz(a,h)anthracene	33 - 120	47	33 - 120	25
	Benzo(g,h,i)perylene	32 - 120	48	29 - 120	34
	Nitrobenzene-d5	48 - 130		48 - 130	
	2-Fluorobiphenyl	47 - 120		47 - 120	
	Terphenyl-d14	33 - 120		33 - 120	
Soil	1,4-Dioxane	6 - 120	20	15 - 120	53
	Naphthalene	55 - 120	20	56 - 120	36
	1-Methylnaphthalene	54 - 120	20	54 - 120	33
	2-Methylnaphthalene	54 - 120	20	53 - 120	32
	Acenaphthylene	43 - 120	20	45 - 120	31
	Acenaphthene	47 - 120	20	43 - 120	51
	Fluorene	48 - 120	20	43 - 120	38
	Phenanthrene	44 - 120	20	19 - 134	72
	Anthracene	40 - 120	20	31 - 125	58
	Fluoranthene	40 - 120	20	7 - 144	66
	Pyrene	44 - 120	20	10 - 153	71
	Benzo(a)anthracene	40 - 120	20	16 - 128	51
	Chrysene	44 - 120	20	21 - 129	60
	Benzo(b)fluoranthene	41 - 120	20	12 - 138	62
	Benzo(k)fluoranthene	40 - 120	20	16 - 131	47
	Benzo(a)pyrene	40 - 120	20	22 - 131	51
	Indeno(1,2,3-cd)pyrene	40 - 120	20	9 - 120	49
	Dibenz(a,h)anthracene	40 - 120	20	13 - 120	47
	Benzo(g,h,i)perylene	40 - 120	20	2 - 120	52
	Nitrobenzene-d5	46 - 120		46 - 120	
	2-Fluorobiphenyl	53 - 120		53 - 120	

Matrix	Analyte	LCS/LCSD Recovery	RPD	MS/MSD Recovery	RPD
	Terphenyl-d14	53 - 127		53 - 127	

C&T internal list numbers: 84002, 84003

Appendix B: Waste Management Plan

FINAL
WASTE MANAGEMENT PLAN

REMEDIAL INVESTIGATION
DRMO SOUTH AND CTA NORTH

FORMER MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

November 2013

Prepared for:

United States Department of the Navy
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FINAL
WASTE MANAGEMENT PLAN

REMEDIAL INVESTIGATION
DRMO SOUTH AND CTA NORTH

FORMER MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

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Approved by:

_____ Date: 11/15/2013

Hoa Voscott, PE
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ARCADIS U.S., Inc.

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NOVEMBER 2013

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ATTACHMENTS

Attachment 1: Waste Inventory Log
Attachment 2: Waste Storage Inspection Form

ACRONYMS

ARCADIS	ARCADIS U.S., Inc.
BRAC	Base Realignment and Closure
CCR	California Code of Regulations
CFR	Code of Federal Regulations
DOT	Department of Transportation
DTSC	Department of Toxic Substances Control
IDW	investigation derived waste
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PMO	Program Management Office
PPE	personal protective equipment
RCRA	Resource Conservation and Recovery Act
SVOC	semivolatile organic compound
T&D	Transportation and Disposal
TCLP	Toxicity Characteristic Leaching Procedure
TPH	total petroleum hydrocarbons
TSDF	treatment, storage, and disposal facility
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound
WMP	Waste Management Plan

1.0 INTRODUCTION

ARCADIS U.S., Inc. (ARCADIS) has prepared this Waste Management Plan (WMP) for remedial investigation (RI) field activities at the Defense Reutilization and Marketing Office (DRMO) South and the Crane Test Area (CTA) North, located at the former Mare Island Naval Shipyard (MINS) in Vallejo, California. This document is submitted as part of the document titled Remedial Investigation Work Plan for the Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California (ARCADIS 2013), hereinafter referred to as the Work Plan. Specific details of the RI field activities are presented in the Work Plan.

This WMP describes the protocols that ARCADIS will implement for collecting, handling, packaging, transporting, and disposing of investigation-derived waste (IDW) for field activities to be performed as part of the RI. The purpose of this WMP is to ensure that waste generated during RI field activities is managed and disposed of safely and in accordance with applicable laws and regulations. This WMP shall be reviewed by field personnel prior to field activities. A copy of this WMP shall be maintained at the project site at all times throughout the duration of field efforts.

2.0 STAFF ORGANIZATION AND RESPONSIBILITIES

Waste management, characterization, sampling, stockpiling, and storage will be performed by ARCADIS, the primary contractor. ARCADIS will assign a Transportation and Disposal (T&D) Coordinator to oversee the T&D-related activities at DRMO South and CTA North. The ARCADIS Field Team Leader will oversee the on-site T&D activities, and the T&D Coordinator will manage the offsite T&D activities. A representative of the Navy will sign all waste disposal manifests as the Generator, provide the appropriate United States Environmental Protection Agency (USEPA) identification number, and perform other related functions required of a waste generator. The waste receiving facility will provide a manifest for each waste stream. Table 2-1 outlines the Site Manager and T&D Coordinator responsibilities during T&D activities. These responsibilities are further described in the following sections of this WMP, and field forms are located in Attachment 1 and Attachment 2.

Table 2-1: Transportation & Disposal Activities and Responsibilities

Field Team Leader Responsibilities	T&D Coordinator Responsibilities
Step 1: Waste accumulation and storage	
Mark and label waste containers. Prepare and maintain Waste Inventory Logs. Inspect storage areas; prepare and maintain Waste Storage Inspection Forms weekly. Ensure waste accumulation times do not exceed regulatory thresholds.	Provide marking and labeling requirements for containers. Monitor Waste Inventory Logs. Monitor Waste Storage Inspection Forms.
Step 2: Waste analysis	
Assist the Project Chemist in retrieving waste characterization samples. Ship samples to laboratory and disposal facilities in accordance with Department of Transportation (DOT) requirements. Provide analytical data to the T&D Coordinator.	Determine waste characterization and disposal facility analysis requirements. Provide waste characterization and disposal facility analysis requirements to the Project Chemist. Determine if samples are regulated by DOT, and provide DOT requirements to the Project Chemist.
Step 3: United States Environmental Protection Agency (USEPA)/Department of Toxic Substances Control (DTSC) waste characterization and DOT waste classification	
Provide site history and waste generation sources to the T&D Coordinator. Verify accuracy of waste labels.	Characterize and classify each waste stream.

Field Team Leader Responsibilities	T&D Coordinator Responsibilities
Step 4: Selection of T&D subcontractors	
Provide the T&D Coordinator with the project schedule.	Determine T&D options. Prepare a purchase order requisition package. Evaluate bidding vendors. Recommend subcontractors.
Step 5: Waste manifest package preparation	
Obtain generator approval and signatures. Submit to the disposal facility and track approval time.	Prepare disposal facility waste profiles, land disposal restriction forms, and template manifests. Obtain disposal facility letters of commitment.
Step 6: Waste shipment and tracking	
Schedule the shipment. Inspect transportation containers, vehicles, and drivers. Obtain generator and transporter signatures on each manifest and land disposal restriction form.	Prepare manifests and land disposal restriction forms. Distribute copies of executed manifests. Track shipment to the disposal facility. Obtain signed manifests from the disposal facility.
Step 7: Waste treatment and disposal	
No action required.	Obtain certificates of disposal/treatment and weight tickets.

3.0 INVESTIGATION-DERIVED WASTE GENERATION

RI field activities at DRMO South and CTA North that will generate IDW include temporary monitoring well installation, soil and groundwater sampling, decontamination activities, and sample packaging and shipment. The following three types of IDW are expected to be generated during field activities:

- **Soil IDW** in the form of excess soil cuttings will be generated during soil coring and sampling activities, as well as during the installation of temporary groundwater monitoring wells.

- **Liquid IDW** in the form of excess groundwater will be generated during temporary groundwater monitoring well installation and development, and groundwater sampling. Liquid IDW will also be generated during decontamination activities.

- **Personal Protective Equipment (PPE), Disposable Equipment, and Administrative Refuse** such as nitrile gloves, disposable sampling equipment, paper towels, tape, sheeting, packaging material, etc. will be generated throughout RI field activities.

These wastes may contain Resource Conservation and Recovery Act (RCRA) listed hazardous wastes, materials exhibiting RCRA hazardous characteristics, or target contaminants above background levels. ARCADIS will make every effort to minimize the overall generation of IDW while maintaining full integrity of the project data quality objectives. Efforts to minimize the generation of IDW include, but are not limited to, the following:

- Combine similar wastes throughout an investigation area in a single container.

- Select appropriately sized containers for waste. For example, use 55-gallon drums for large volumes of waste but select containers with less capacity for small volumes of waste.

- Tightly pack solid waste into containers to minimize the number of containers required.

- Use nonhazardous or less hazardous substances whenever possible.

It is not anticipated that hazardous materials will be used during the RI activities at DRMO South and CTA North. Should hazardous materials be used, copies of the corresponding Material Safety Data Sheets will be provided, and hazardous materials will be maintained by the Safety Department. Hazardous materials will be stored and labeled in accordance with the project specifications. Waste derived from the use of hazardous materials will be stored near the point of generation for up to a total quantity of 1 quart of toxic waste or 55 gallons of hazardous waste. Any volume that exceeds those quantities will be moved to a hazardous waste permitted area within three days.

4.0 WASTE HANDLING

IDW will be handled in accordance with local, state, and federal regulations.

4.1 Soil Investigation-Derived Waste

Soil IDW will be generated during soil sampling and temporary well installation activities and may contain the following chemicals: volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs) including polycyclic aromatic hydrocarbons (PAHs), metals, polychlorinated biphenyls (PCBs), pesticides, and total petroleum hydrocarbons (TPH; ARCADIS 2013). Soil IDW will be handled using the following procedure:

- Place soil cuttings in DOT-approved 55-gallon drums.
- Fill drums until the lesser of 6 inches or 5 percent of head space is obtained to allow for waste expansion during freezing or heating.
- Close and secure open-topped drums with the bolt at the end of each day and when full.
- Label and number the drum using the procedures described in Section 5.1.1.
- Stage the drum at the point of generation. When full, or when usage of the drum is complete, move the drum to the common secured storage area while awaiting analysis and characterization. The location of the secured storage area will be established with the Caretaker Site Office representative and/or Navy Resident Officer in Charge of Construction prior to the start of field work.
- Conduct waste analysis and characterization using the procedures outlined in Section 6.
- Following waste characterization and profiling, transfer containers to the T&D Coordinator for final offsite disposal at an appropriate licensed disposal facility.

4.2 Liquid Investigative Derived Waste

Liquid IDW generated during groundwater well installation, development, and sampling activities may contain the following chemicals: VOCs, SVOCs including PAHs, metals, PCBs, pesticides, and TPH (ARCADIS 2013).

Liquid IDW generated during equipment decontamination activities may contain chemicals from groundwater and/or soil, depending on what media the equipment was used to sample. Liquid IDW generated during decontamination may contain VOCs, SVOCs including PAHs, metals, PCBs, pesticides, and TPH.

Liquid IDW will be handled using the following procedure:

1. Place liquid IDW in a holding tank and/or DOT-approved, 55-gallon drum.

2. Close and secure open-topped drums with the bolt at the end of the day and when full.
3. Label and number the drum using the procedures described in section 5.1.1.
4. Stage the drum at the point of generation. When full, or when usage of the drum is complete, move the drum to the common secured storage area while awaiting analysis and characterization.
5. Conduct waste analysis and characterization on drums using the procedures outlined in Section 6.
6. Following waste characterization and profiling, transfer containers to the T&D Coordinator for final offsite disposal at an appropriate licensed disposal facility.

4.3 PPE, Disposable Equipment, and Administrative Refuse

PPE, disposable equipment, and administrative refuse will be sorted and disposed of as general municipal refuse or recyclables. Disposable equipment and gloves will be rinsed prior to disposal, and rinse water will be considered liquid IDW.

In the event that PPE, disposable equipment, or administrative refuse come into contact with contaminated materials, they will be thoroughly decontaminated and disposed of using the following procedure:

1. Scrub and rinse contaminated waste with a solution of potable water and Alconox until all visible residue and visible potential contaminants are removed.
2. Rinse waste in two separate containers of potable water.
3. Place decontaminated waste into a garbage bag, seal the bag, and dispose of it in an on-site trash receptacle.

If PPE, disposable equipment, or administrative refuse cannot be successfully decontaminated (e.g., it is too large to decontaminate or has come in contact with free product), they will be handled using the following procedure:

1. Separate the waste from the municipal waste stream and place it in a DOT-approved, 55-gallon steel drum.
2. Close and secure open-topped drums with the bolt at the end of the day and when full.
3. Label and number the drum using the procedures described in Section 5.1.1.
4. Stage the drum at the point of generation. When full, or when usage of the drum is complete, move the drum to the common secured storage area while awaiting analysis and characterization.
5. Conduct waste analysis and characterization on drums using the procedures outlined in Section 8.
6. Following waste characterization and profiling, transfer containers to the T&D Coordinator for final offsite disposal at an appropriate licensed disposal facility.

5.0 WASTE ACCUMULATION & INTERIM STORAGE

5.1 Waste Management

Inventory and tracking of waste containers (drums, bags, etc.) containing IDW will be managed through a three-step process. The process includes proper labeling, assignment of identification numbers, and accurate and timely completion of the Waste Inventory Log included as Attachment 1.

5.1.1 Labeling

Containers used to store potentially hazardous IDW will be labeled in the field prior to filling the container. Each container will be covered and sealed daily before field personnel leave the site. No unlabeled containers will be used or left on site. Containers will have a stick-on label on the side and lid, and permanent paint markers will be used to mark the labels. Container identification numbers will be assigned by ARCADIS or by the Navy. The container will include the following information:

- Dates of first and last use
- Site address
- Facility address, if applicable
- Well identification, if applicable
- Waste type (e.g., soil cuttings or wastewater)
- Generator name and contact information
- A unique identification number (to be assigned by ARCADIS or the Navy using the procedures in Section 5.1.2)

If applicable, “Awaiting Analysis” will be written on each label.

5.1.2 Container Identification Number

All containers of IDW will be numbered to aid in waste management during fieldwork. Identification numbers assigned by ARCADIS will use the following numbering convention:

- The first two digits of the container identification number will identify the study as RI.
- The following four or five digits of the container identification number will identify the site from which the IDW was derived (i.e., DRMOS or CTAN).

- The last four digits will begin with 0001 and will be carried forward sequentially for each container at each site.

For example, if three containers of IDW are generated from DRMO South, they would be labeled RI-DRMOS-0001, RI-DRMOS-0002, and RI-DRMOS-0003.

5.1.3 Waste Inventory Log

The Waste Inventory Log will be maintained onsite by the ARCADIS Field Team Leader and will contain the following information:

- Unique container identification number
- Site ID
- Container type (e.g., drum, bag)
- Container contents (e.g., soil cuttings, groundwater, decontamination water, contaminated PPE, contaminated sampling equipment, contaminated administrative refuse)
- Date the container was filled
- Date the waste characterization sample was collected
- Date of transfer to the storage area, if applicable
- Date by which analytical results must be transmitted to the Navy
- Date on which analytical results were received by the Navy
- Waste determination decision
- Date on which container was transferred to the Treatment, Storage, and Disposal Facility (TSDF)

An example Waste Inventory Log is included as Attachment 1.

Trained ARCADIS staff or subcontractor team members will track the IDW containers daily using the Waste Inventory Log and will document movement of project IDW in a field logbook. Staff will be thoroughly familiar with proper waste handling and emergency procedures during normal facility operations and during emergencies. The Waste Inventory Log will be returned to the ARCADIS Field Team Leader at the end of each work day.

5.2 Storage Area

IDW will be temporarily stored in a selected onsite storage area. Drums will be labeled using the procedures described in Section 5.1.1 prior to placement in the storage area. ARCADIS will segregate and accumulate wastes into the categories outlined in Table 5-1. The ARCADIS Field Team Leader will ensure wastes from different sources are additionally segregated by each individual source. The T&D Coordinator will then review available information and determine whether the wastes from different sources can be commingled for cost and handling efficiency.

The Field Team Leader will inspect IDW containers and storage weekly per Title 22 of the California Code of Regulations (CCR) §66265.174 and will document the results using the Waste Storage Inspection Form (included as Attachment 2). The inspection will note the container number, storage location, container origin date, container condition, and any comments. Where a hazardous situation is imminent or exists, corrective action will be taken immediately.

Table 5-1: Waste Accumulation Methods and Times

Waste	Accumulation Method	Maximum Accumulation Time
Soil Class 1 (Hazardous) contaminated with one or more chemicals[1]	DOT-approved, 55-gallon drums	90 days[3]
Soil Class 2 (Nonhazardous, no regulated contaminants)	DOT-approved, 55-gallon drums	None
Liquid IDW (Hazardous) contaminated with one or more chemicals[2]	DOT-approved, 55-gallon drums	90 days[3]
Liquid IDW (Nonhazardous, no regulated contaminants)	DOT-approved, 55-gallon drums	None
Contaminated PPE, disposable equipment, and administrative refuse	DOT-approved, 55-gallon drums	90 days[3]
Non-contaminated or decontaminated PPE, disposable equipment, and administrative refuse	Plastic bags	None

[1]Potential chemicals in Soil IDW include VOCs, SVOCs including PAHs, metals, PCBs, pesticides, and TPH.
[2]Potential chemicals in Liquid IDW include VOCs, SVOCs including PAHs, metals, PCBs, pesticides, and TPH.
[3]Per 22 CCR §66262.34

6.0 WASTE PROFILING

ARCADIS will profile the waste to determine whether it is hazardous and to determine the proper transport and disposal methods with Navy approval.

6.1 Waste Analysis Requirements

The Field Team Leader will retrieve representative samples for waste characterization and may retrieve additional grab samples for comparison to applicable EPA/Department of Toxic Substances Control (DTSC) land disposal restrictions.

IDW in the form of PPE, disposable equipment, or administrative refuse that cannot be successfully decontaminated due to size or nature of the waste, or because the waste has been exposed to free product, will be profiled to assess proper disposal options.

Soil IDW and liquid IDW will require profiling to assess proper disposal options. Soil and liquid IDW samples will be collected and analyzed for the following chemicals anticipated to be present in site media and as required by the disposal facility:

- California Title 22 metals
- SVOCs (including PAHs)
- VOCs
- PCBs
- Pesticides
- TPH

Table 6-1 summarizes the anticipated sample types and analytical methods necessary for proper waste characterization and waste disposal acceptance. If necessary the following analyses will also be performed for soil IDW:

- Soluble Threshold Limit Concentrations by California Waste Extraction Test
- Toxicity Characteristic Leaching Procedure (TCLP)

Table 6-1: Waste Sample Types and Analyses

Waste Characterization	Sample Type	Analytical Methods
Soil Class 1 (Hazardous) Soil Class 2 (Nonhazardous)	One 4-point composite sample	Method 6010B/7471A for California Title 22 metals Method 8015B for TPH as diesel and motor oil Method 8270C for SVOCs Method 8081A for pesticides Method 8082 for PCBs
	Grab sample using EnCore® sampling device	Method 8260B for VOCs Method 8015B for TPH as gasoline
Liquid IDW (Hazardous) Liquid IDW (Nonhazardous)	Grab sample using bailer	Method 6010B/7470A for California Title 22 metals Method 8015B for TPH Method 8270C for SVOCs Method 8081A for pesticides Method 8082 for PCBs Method 8260B for VOCs
PPE, disposable equipment, administrative refuse	Not Applicable Non-contaminated and decontaminated PPE, disposable equipment, and administrative refuse will be recycled or disposed of in the municipal waste stream. If IDW cannot be properly decontaminated, analytical data from the environmental samples that generated the IDW will be used to determine potential disposal options.	Not Applicable

6.2 Waste Characterization and Classification

ARCADIS will assign the appropriate USEPA waste codes to each waste stream in accordance with the criteria listed in 40 Code of Federal Regulations (CFR) Part 261

Subpart B for identifying the characteristics of hazardous wastes (40 CFR 261 Subpart C) and for listing hazardous wastes (40 CFR 261 Subpart D), as well as applicable criteria in Title 22 of the CCR in cases where it is stricter than Federal regulation. Two sources of data may be used to support the waste characterization:

- **Analytical Data** from soil samples and groundwater samples may be used to characterize the soil and liquid IDW generated during soil coring, temporary monitoring well installation, or other field activities associated with the sample collection.

- **Historical Knowledge** of the processes which generated the wastes disposed of in an investigation area may be used to identify possible listed hazardous waste codes that may apply to IDW.

Containerized soil and liquid IDW will undergo laboratory analysis, and any compounds with concentrations exceeding regulatory limits will be identified for: RCRA characteristics (ignitability, corrosivity, reactivity, toxicity); and exceedance of TCLP threshold values for concentrations in excess of regulatory limits for total concentration.

Hazardous IDW generated during this project is anticipated to be assigned the "D" USEPA waste code, which indicates that it exhibits one of the four characteristics of hazardous waste: ignitability, corrosivity, reactivity, or toxicity (40 CFR §261.20-20).

These properties are quantifiable by standardized and available testing methods that can be found in SW-846: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. If the detected level exceeds the regulated level or if the waste exhibits any of the three characteristics (ignitability, corrosivity, reactivity), the waste will be assigned the appropriate corresponding D-code.

The preliminary characterization and classification of IDW generated during field activities is outlined in Table 6-2. Additional waste characterization may be performed to determine the exact disposal volumes and requirements.

Table 6-2: Investigation-Derived Waste Characterization and Classification

Waste Stream	USEPA/DTSC Characterization	DOT Classification
Soil Class 1 (Hazardous)	Hazardous	Class 9 (Miscellaneous Hazardous Material)
Soil Class 2 (Non-hazardous, no regulated contaminants)	Nonhazardous	Not regulated
Liquid IDW (Hazardous)	Hazardous	Class 9 (Miscellaneous Hazardous Material)
Liquid IDW (Non-hazardous, no regulated contaminants)	Nonhazardous	Not regulated
Non-contaminated or decontaminated PPE, disposable equipment, and administrative refuse	Nonhazardous	Not Applicable
Contaminated PPE, disposable equipment, and administrative refuse	Hazardous	Class 9 (Miscellaneous hazardous Material)

7.0 TRANSPORTATION AND DISPOSAL

Following characterization and profiling of the waste, the T&D Coordinator will prepare a manifest package for each waste stream for offsite disposal or recycling. Disposal of RCRA wastes, or recycling of such wastes, will occur only at USEPA-approved facilities. Each manifest package will be signed by a Navy representative as the Generator. All subcontractors utilized for transportation and/or disposal will possess all applicable state and federal permits, certifications, and licenses required to legally perform the requested tasks.

7.1 Nonhazardous Waste Disposal

ARCADIS will implement the following procedure for offsite nonhazardous IDW disposal:

1. The T&D Coordinator will fill out the Generator Waste Profile Form for the appropriate selected disposal facility and include analytical data from waste characterization, showing the maximum detection of each constituent.
2. The T&D Coordinator will then contact the disposal facility discussing the volume of waste planned for disposal, source of waste, and brief interpretation of the waste characterization data.
3. The T&D Coordinator will transmit the disposal facility Generator Waste Profile Form and analytical data to the disposal facility.
4. The T&D Coordinator will then fill out a nonhazardous waste manifest to ship Non-RCRA, Non-California Hazardous Waste.
5. Upon the disposal facility's approval, the Field Team Leader will schedule the waste material transportation by a certified waste transporter and its disposal at the selected disposal facility.

7.2 Hazardous Waste Disposal

ARCADIS will implement the following procedure for offsite hazardous waste disposal:

1. The T&D Coordinator will prepare a waste profile using the waste characterization data, along with the analytical data for reference, and submit it to the selected TSDF for approval.
2. Following TSDF approval to accept the waste and 3 to 5 days prior to shipment, the T&D Coordinator will prepare and complete a hazardous waste manifest using USEPA Form 8700-22 and, if necessary, the USEPA Continuation Form 8700-22A. The hazardous waste manifest will be sent to the Navy for pre-approval. The Field Team Leader will then coordinate the transportation schedule with Navy personnel designated to sign the manifest.
3. The Field Team Leader will then schedule waste material transportation by a certified waste transporter and its disposal at an appropriate TSDF.
4. The T&D Coordinator will prepare six copies of the hazardous waste manifest for distribution. The Field Team Leader will obtain a handwritten signature from the

initial transporter with the date of acceptance. ARCADIS will retain one copy of the signed manifest (22 CCR §66262.40[a]) and make photocopies. ARCADIS will then send four copies with the transporter. ARCADIS will submit two signed copies to the Navy Environmental Compliance Manager for their records and submission to DTSC, and retain one copy for the facility files. The transporter will keep one copy and give three copies to the TSDF. The TSDF will then return one signed copy to the Mare Island Facility, one signed copy to DTSC, and retain one copy for their files. The TSDF should return the signed copy to the Mare Island Facility within 35 days from its receipt of the waste; otherwise if the TSDF does not return the manifest within 45 days, the Navy generator must file an exception report with the DTSC (22 CCR §66262.42).

8.0 DOCUMENTATION AND RECORDKEEPING

In accordance with 22 CCR §66262.40, the Navy generator will maintain original copies of the signed manifests and waste characterization date pages for three years from the date the waste was accepted by the initial transporter. ARCADIS will keep IDW-related field logbook entries, Waste Inventory Logs, and Waste Storage Inspection Forms on file for the duration of the project. At the conclusion of the project, the Navy will maintain the completed original Waste Inventory Log and Waste Storage Inspection Forms as part of the Mare Island Facility operating record, which will provide evidence of compliance with this WMP.

9.0 REFERENCES

ARCADIS U.S., Inc. (ARCADIS). 2013. Draft Remedial Investigation Work Plan for the Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California. July.

Attachment 1: Waste Inventory Log

HAZARDOUS WASTE INVENTORY LOG

Container ID[1]	Site ID[2]	Container Type	Contents[3]	Fill Date[4]	Sample Date[5]	Transfer Date[6]	Analytical Results Due Date[7]	Analytical Results Receipt Date[8]	Waste Determination[9]	TSDF Transport Date[10]

[1] See Section 5.1.2 of the Waste Management Plan for Container ID numbering schemes.
[2] Site from which the container originated
[3] Example descriptions include soil cuttings, groundwater, decontamination water, contaminated PPE, contaminated sampling equipment, contaminated administrative refuse, etc.
[4] Date container was filled
[5] Date waste characterization sample(s) collected from container
[6] Date container was transferred to storage area
[7] Date analytical results are due to the Navy (Mare Island Facility)
[8] Date analytical results were received by the Navy (Mare Island Facility)
[9] Waste classification (e.g. hazardous, non-hazardous) based on waste characterization
[10] Date container was transferred to the Treatment, Storage, and Disposal Facility (TSDF)

Attachment 2: Waste Storage Inspection Form

HAZARDOUS WASTE STORAGE AREA WEEKLY INSPECTION CHECKLIST

Inspector Name: _____ **Date:** _____ **Time:** _____

Location of Inspection: _____ **Total Number of Containers:** _____

		YES	NO
1.	Is the area free of debris and other materials?	☐	☐
2.	Is the ground clean and dry?	☐	☐
3.	Are container tops free of spillage?	☐	☐
4.	Is the area free of spills or leaks?	☐	☐
5.	Are all of the containers in good condition? (free of dents and corrosion, not bulging, or otherwise deteriorating?)	☐	☐
6.	Are all containers properly closed?	☐	☐
7.	Are containers labeled with hazardous waste labels?	☐	☐
8.	Is the following information on the labels filled out?		
	Generator name and address	☐	☐
	Accumulation start date	☐	☐
	Contents	☐	☐
	Physical state	☐	☐
	Hazardous properties	☐	☐
9.	Is the information on the labels legible?	☐	☐
10.	Have wastes been disposed of within the allowable accumulation time?	☐	☐
11.	Are the containers compatible with their contents?	☐	☐
12.	Are incompatible wastes stored separately?	☐	☐
13.	Is there adequate aisle space?	☐	☐

Describe any observations for items checked 'NO'. _____

_____

_____

_____

_____

Corrective actions required. _____

_____

_____

_____

*** Inspections must be conducted on a weekly basis**

*** Maintain checklist as documentation of this requirement**

*** Inspection program must meet requirements of 22 CCR §66265.174**

Appendix C: Environmental Protection Plan

FINAL
ENVIRONMENTAL PROTECTION PLAN

REMEDIAL INVESTIGATION
DRMO SOUTH AND CTA NORTH

FORMER MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

November 2013

Prepared for:

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FINAL
ENVIRONMENTAL PROTECTION PLAN

REMEDIAL INVESTIGATION
DRMO SOUTH AND CTA NORTH

FORMER MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

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Approved by:

_____ Date: ____11/15/2013____

Hoa Voscott, PE
Project Manager
ARCADIS U.S., Inc.

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NOVEMBER 2013

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FIGURES

Figure 1 Site Location Map

ACRONYMS

APP/SSHP	Accident Prevention Plan/Site Safety and Health Plan
ARCADIS	ARCADIS U.S., Inc.
BMP	best management practices
CDFW	California Department of Fish and Wildlife
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CTA	Crane Test Area
DRMO	Defense Reutilization and Marketing Office
DTSC	Department of Toxic Substances Control
EPP	Environmental Protection Plan
MINS	Mare Island Naval Shipyard
mph	miles per hour
NTCRA	Non-Time Critical Removal Action
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
RPM	Remedial Project Manager
RI	remedial investigation
Water Board	Regional Water Quality Control Board
SI	site inspection
SL	screening level
TPH	total petroleum hydrocarbons
TPH-d	total petroleum hydrocarbons as diesel
TPH-mo	total petroleum hydrocarbons as motor oil
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service

1.0 INTRODUCTION

1.1 Purpose and Scope

ARCADIS U.S., Inc. (ARCADIS) has prepared this Environmental Protection Plan (EPP) for remedial investigation (RI) field activities at the Defense Reutilization and Marketing Office (DRMO) South and the Crane Test Area (CTA) North, located at the former Mare Island Naval Shipyard (MINS) in Vallejo, California. This document is submitted as part of the document titled Remedial Investigation Work Plan for the Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California (ARCADIS 2013a), hereinafter referred to as the Work Plan. Specific details of the RI field activities are presented in the Work Plan.

The objective of this EPP is to describe environmental protective measures to control field activities during the RI. This EPP describes environmental compliance procedures as well as regulatory, procedural, and training requirements associated with RI fieldwork. This EPP shall be reviewed by all site field personnel prior to field activities, and a copy of this EPP shall be maintained at the site at all times. This document will be updated if the scope changes or environmental regulations are revised.

1.2 Site Description

DRMO South is positioned within the north-central portion of MINS, south of Dump Road and the DRMO site and west of Azuar Drive (Figure 1). A large portion of the site is undeveloped, open space area while the remainder includes industrial area and vehicle parking lots. The CTA North is an undeveloped, open space area also located in the north-central portion of MINS. The boundaries of CTA North are roughly defined as the upland area north of Dump Road and west of Azuar Drive (Figure 1). The CTA is not included within the CTA North boundaries; the former CTA was transferred to the City of Vallejo, which then transferred the CTA to the Mare Island redeveloper, Lennar Mare Island, in 2001.

The topography at DRMO South and CTA North is relatively flat, and ecological habitat consists of uplands with ruderal vegetation and adjacent non-tidal wetlands containing a variety of vegetation including cattails, pickleweed, and sedges.

Per the current reuse plan, future uses of DRMO South include light industrial and open space; and CTA North will remain undeveloped open space (City of Vallejo 2007).

1.3 Site History and Condition

DRMO South is currently vacant with several unused buildings including Buildings 275, 961, 965, and 831. Removal actions conducted in the neighboring DRMO site between 2005 and 2008 (non-time-critical removal action [NTCRA]) and between 2009 and 2010 (petroleum corrective action [PCA]) included the excavation of small areas of the northwest and eastern boundaries of DRMO South.

No buildings are or were previously located within the boundaries of CTA North, which comprises undeveloped open space. Between 2009 and 2010, petroleum-impacted soil was excavated from an area within and immediately adjacent to CTA North as part of the PCA. During the PCA, excavation areas with bottom or confirmation samples exceeding the screening levels (SLs) were over-excavated and resampled with the exception of four sidewall samples located at the northern limit of the excavation area within CTA North (Weston 2010). These samples exhibited total petroleum hydrocarbon (TPH) concentrations above the Tier 2 SLs but were not over-excavated (Weston 2010).

Site inspections (SIs) were conducted at DRMO South and CTA North in October and November 2012 in response to a letter from the California Department of Toxic Substances Control (DTSC) requesting additional information and/or data to address the possibility that affected soil and groundwater similar to that observed at the CTA (on Lennar Mare Island property) may also be present on the adjacent Navy property (DTSC 2009). The Navy also identified DRMO South, located in the vicinity of CTA North, for further investigation based on the findings of the Internal Draft Investigation Area H2 RI Report (Tetra Tech EM, Inc. 2000). Investigation Area H2 included DRMO South as well as portions of CTA North. Results of the 2012 SIs demonstrated that chemicals including metals, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), TPH as diesel (TPH-d), and TPH as motor oil (TPH-mo) were present in soil at concentrations exceeding human health and/or ecological screening criteria. Chemicals detected in groundwater at concentrations exceeding human health and/or ecological screening criteria included metals, pesticides, volatile organic compounds, and PAHs.

1.4 Scope and Objectives

The objectives of the RI are to characterize site conditions, evaluate the nature and extent of affected soil and groundwater, refine the conceptual site model, and assess potential risk to human health and the environment. Information obtained during this process will guide future decisions made regarding DRMO South and CTA North. A detailed description of the field activities is included in the Work Plan.

1.5 Protection of Selected Features

Based on the locations of and existing conditions at DRMO South and CTA North, the following categories of controls will be required during the RI: land resource controls, air quality controls, surface water quality controls, and spill prevention and response measures.

1.6 Plan Implementation

1.6.1 Project Organization

Investigation of DRMO South and CTA North sites is being conducted per the Navy's direction. The Department of Defense has the authority to undertake Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) response actions under Title 42 United States Code Part 9604 (42 USC 9604), 10 USC 2708, and Federal Executive Order 12580. The Navy is the lead agency responsible for the cleanup effort, the

DTSC is the lead regulatory agency overseeing Installation Restoration Program activities with support from the San Francisco Bay Region Regional Water Quality Control Board (Water Board), United States Environmental Protection Agency (USEPA), United States Fish and Wildlife Service (USFWS), and California Department of Fish and Wildlife (CDFW).

ARCADIS will be responsible for implementing and monitoring compliance with this EPP.

1.6.2 Briefings

All personnel conducting work associated with the RI, including subcontractor employees, will receive briefing on the contents of this EPP. Briefings will include education on detection and prevention of pollution onsite, as well as procedures for responding to potential spills, should they occur. Briefings on biological avoidance measures will also be conducted as described in Section 2.3

2.0 LAND RESOURCES

Prior to initiating field work, ARCADIS will locate and mark existing utilities and will identify and delineate the authorized work and staging areas.

2.1 Historical, Archeological, and Cultural Resources

There are no historical, archeological, or cultural resources currently identified within DRMO South or CTA North. If such resources are discovered, work in the area will stop and the Navy Remedial Project Manager (RPM) will be notified. ARCADIS employees and all subcontractors working under ARCADIS will record, report, and preserve the findings in accordance with federal Protection of Archeological Resources regulations.

2.2 Existing Vegetation

No significant vegetation disturbance is expected during the RI soil and groundwater sampling activities.

2.3 Fish and Wildlife/Threatened, Endangered, and Sensitive Species

The planned RI sampling activities will be conducted adjacent to non-tidal wetland areas that are considered potential habitat for wildlife species including the salt marsh harvest mouse, which is a state- and federal-listed endangered species. Monitoring for the California Clapper Rail and California Black Rail will be conducted in conjunction with salt marsh harvest mouse monitoring even though the California Clapper Rail and California Black Rail are less likely to be encountered at DRMO South and CTA North.

A qualified biological monitor approved by the USFWS and the CDFW will be responsible for implementing the required biological avoidance, minimization, and mitigation measures. These measures will conform with those presented in the Final PA/SI Work Plan for DRMO South and CTA North (Trevet 2012) and will include the following:

- The biological monitor will have the authority to stop work if deemed necessary for any reason to protect salt marsh harvest mice (or any other state or federal "Threatened or Endangered" or "Fully Protected" species, including rails, rare plants, and birds protected by the Migratory Bird Treaty Act of 1918 (16 USC 703-712).

- Prior to work, all contractor and subcontractor personnel will be required to attend a Biological Resource Education Program briefing conducted by the biological monitor. This briefing will: (1) review the sensitive biological resources [e.g., salt marsh harvest mouse, jurisdictional wetlands] potentially located within the work area; and (2) identify all protective measures to be implemented and complied with to ensure that these resources are not affected by work activities. New employees will attend a briefing by the biological monitor prior to participating in work activities.

- If site work is scheduled to be conducted between February 1 and September 15 (i.e., the nesting season), the biological monitor will conduct a visual inspection for nesting birds. The visual inspection will cover all staging and storage areas, transportation routes, work areas, and soil stockpile areas, and will be completed within a reasonable time prior to the commencement of any project activities. If work is already occurring in the above-listed areas when nesting season starts, a nesting visual inspection will not be required. If work is stopped for a longer than reasonable period of time, as determined by the qualified biologist, during the nesting season, staging and storage areas, work areas, and soil stockpile areas will be reassessed before work activities are resumed.

- The site biological monitor will inspect the work area and natural habitats occurring within a reasonable distance of the work area to identify active nests. If no active nests are found, no further mitigation will be conducted. If nesting birds are found during the visual inspections, the locations will be noted in the field log book, appropriate avoidance measures will be recommended by the biologist, and the Navy will submit the report to the USFWS and CDFW. If a qualified biologist, in coordination with the Navy, USFWS, and CDFW, determines that the project activity will not likely adversely affect the active nest, the project may proceed. The qualified biologist will monitor the activity of the nesting birds (including rails) during work activities. If the birds behave normally, the biologist will monitor them twice per week to ensure that the status has not changed. If the birds change their behavior as a result of work activities, the biologist will continue to monitor the birds as work is modified until the birds act normally. The biologist will then monitor the birds twice per week to ensure that the status has not changed.

- If a rare plant species is observed in the field, the location will be noted in the daily report, appropriate avoidance measures will be recommended by the biologist, and the Navy will submit the report to the USFWS and CDFW. If a sampling location is affected by the presence of a rare plant species, the location will be offset so as to avoid the need to remove the rare plant. If this is not feasible and the sample must be collected at that location, the Navy will notify the USFWS and CDFW, and the Navy will coordinate appropriate avoidance and mitigation measures with these agencies.

- Sample locations will be offset as necessary to avoid the removal of wetland vegetation.

- The equipment decontamination area will be located in designated upland staging areas away from non-tidal wetlands.

- Drilling and other heavy equipment will be confined to access routes, designated staging areas, and designated drilling areas. Upland routes covered with pavement, bare ground, or non-native vegetation will be utilized as access routes to and from the designated sampling areas, to the maximum extent practicable.

- No vehicles will require access to the non-tidal wetlands area, and no heavy equipment will be used within 50 feet of the non-tidal wetlands.

There are no plans to collect samples from within 50 feet of the non-tidal wetlands as part of the RI. Sampling locations will remain outside of the 50-foot buffer zone for the non-tidal wetlands unless the regulatory agencies specifically request otherwise or unless extraordinary circumstances arise. If heavy equipment will be used within 50 feet of the non-tidal wetlands, the following additional mitigation measures will be implemented:

- A silt fence will be installed between the work sites and areas of non-tidal wetlands immediately following vegetation removal and before sampling activities begin to prevent the entry of salt marsh harvest mice into cleared areas. The final design and proposed location of the fencing will be reviewed and approved by the USFWS and CDFW prior to the installation. The fencing will be trenched into the ground and backfilled to prevent the mice from moving underneath the fencing. All silt fencing will be installed with stakes facing towards the work site and away from the habitat. The biological monitor will have the ability to make field adjustments to the location of the fencing depending on site-specific habitat conditions.

- The biological monitor will monitor silt fencing periodically during the work day to ensure that it remains an effective barrier to prevent entry of salt marsh harvest mice into work areas. Maintenance of the fencing will be conducted as needed throughout the work period. Any necessary repairs to the fencing will be completed within 24 hours of the initial observation of the damage. Work may continue while the fences are repaired once the qualified biologist has ensured that salt marsh harvest mice have not entered the work area.

- Each day, prior to the initiation of work within 50 feet of non-tidal wetlands, the biological monitor will thoroughly inspect the work area and adjacent habitat areas to determine if salt marsh harvest mice, rails, rare plants, or birds protected by the Migratory Bird Treaty Act are present in these areas, and will remain on site throughout the day while work activities occur in these areas. If a mouse of any species or a rail is observed, then work in the area of the observed species will not be initiated or will be stopped immediately by the biological monitor until the mouse or rail leaves the area of its own volition and the USFWS and CDFW are notified. If the mouse or rail does not leave the intrusive investigation area, work will not be reinitiated until the USFWS and CDFW are contacted and have made a decision on how to proceed with work activities. The biological monitor will direct the contractor on how to proceed accordingly.

- If vegetation removal is required within non-tidal wetlands, the vegetation will be removed under the supervision of a qualified biologist. The qualified biologist will survey the areas for salt marsh harvest mice, rails, rare plants, and birds protected by the Migratory Bird Treaty Act prior to vegetation removal. If a mouse of any species is observed within the areas being cleared of vegetation, the USFWS and CDFW will be notified. Unless otherwise approved by the USFWS and CDFW, the mouse shall be left

alone and allowed to move out of the area of its own volition. Vegetation removal may only begin when no mice are observed.

- If vegetation removal is required within the 50-foot buffer zone, the process will start at the edge farthest from the non-tidal wetlands and will work its way towards the wetlands. This method will provide cover for salt marsh harvest mice and will allow them to move toward the non-tidal wetlands of their own volition as vegetation is being removed. Non-tidal wetland vegetation and any vegetation that needs to be removed for sampling activities within a 50-foot buffer zone around the non-tidal wetlands will be removed using non-motorized hand tools only (mechanized tools are acceptable). A biological monitor will be present during vegetation removal conducted within 50 feet of the non-tidal wetlands. Non-handheld equipment will be limited to the areas where the vegetation has been cut. Personnel may only walk into coastal salt marsh wetlands when accompanied by the USFWS- and CDFW-approved biologist.

2.4 Utilities

ARCADIS or its subcontractor will locate and mark all utilities within the authorized work areas. If a proposed sample location is not accessible due to presence of an underground utility, the sample location will be shifted to an accessible position as close to the originally proposed location as practical.

2.5 Site Access Control

All personnel obtaining access to the sites will utilize a site sign-in sheet. Visitors will be permitted at the site only with the permission of the Site Safety and Health Officer.

2.6 Recycling and Waste Prevention Measures

ARCADIS will integrate recycling, waste-prevention, waste minimization, and energy conservation measures into daily operations at the sites.

When waste is generated, efforts will be made to minimize the extent. Cleanup activities will be implemented both during and upon completion of the RI field work to keep the sites and adjacent properties free from the accumulation of contractor-generated wastes. In accordance with good housekeeping and materials management practices, all waste materials, rubbish, and debris generated during the project will be placed in the appropriate containers (e.g., plastic bags or drums) as soon as practicable and disposed of properly.

Vehicles and heavy equipment will be shut down when not in operation. Upon completion of work, all contractor vehicles and equipment will be removed from the site, and the sites and staging areas will be restored to a condition similar or equal to that existing prior to work.

3.0 AIR QUALITY

The Bay Area Air Quality Management District regulates the standards for fugitive dust emissions and particulate matter emissions. RI field activities are not anticipated to produce significant dust; however, dust control measures may be implemented during fieldwork to mitigate or prevent dust nuisance.

The following summarizes potential dust control measures:

- Construction vehicle speed at the work site will be limited to 15 miles per hour (mph) or less.
- To the maximum extent practicable, construction traffic will be limited to paved areas, and only essential vehicles and equipment will be permitted to enter areas where soil was disturbed.
- Vehicles and equipment will use designated entrances and on-site travel routes.
- Unpaved areas subject to vehicle traffic will be adequately wetted. Water may also be applied to paved areas, if necessary.
- The work area will be adequately wetted prior to any ground disturbance.
- Water spray dust control (i.e., the repeated application of sprayed water or mist) will be implemented during all soil handling activities to reduce the generation of dust.
- The area subject to construction activity will be limited at any one time.
- Equipment and vehicles will be visually inspected and, if necessary, swept free of loose soil and/or washed down prior to leaving the work areas.
- All sidewalks and streets affected by the work will be swept clean.
- When wind speeds exceed 20 mph, soil handling activities will be monitored and suspended if dust is unmanageable.

Air monitoring will be conducted as described in the project-specific Accident Prevention Plan/Site Safety and Health Plan (APP/SSHP; ARCADIS 2013b).

It is anticipated that project vehicles and equipment will generate limited noise during operation. However, the noise levels are not anticipated to disturb workers or surrounding populations.

4.0 WATER QUALITY

The integrity of surface water and groundwater resources at the site will be maintained throughout the RI fieldwork. ARCADIS will control the use and disposal of fuels, oils, or other harmful materials, and precautionary measures will be implemented to prevent these materials from impacting surface water.

4.1 Critical Protected Areas

Non-tidal wetlands are located near the work areas. These wetland areas are more sensitive to chemical and hazardous materials spills, and ARCADIS will take reasonable precaution to prevent the discharge or runoff of hazardous materials into the non-tidal wetlands.

4.2 Stormwater Pollution Prevention

Because the RI activities will disturb an area of less than one acre, the National Pollution Discharge Elimination System General Permit for Stormwater Discharges Associated with Construction Activity is not an applicable or relevant and appropriate requirement. However, appropriate best management practices (BMPs) will be implemented during the RI field activities to minimize the potential for site pollution. These BMPs may include:

- Scheduling field work for the dry season.
- Employing sediment controls (e.g., use of straw wattles and/or straw bale/sandbag barriers to reduce runoff in the event of a storm).
- Regularly inspecting equipment for leaks and immediately cleaning all leaks, drips and other spills, using dry methods if possible.
- Refueling vehicles and equipment in designated fueling areas away from storm drains, using secondary containment and spill protection.
- Maintaining proper material handling, delivery, and storage practices, which include designation of areas for specific activities/items such as material storage, waste collection, decontamination, equipment maintenance/fueling, and sanitary facilities.
- Collecting liquids generated during decontamination, washing, etc., for proper offsite disposal.
- Designating areas for vehicle and equipment cleaning where runoff is not likely to be generated and away from existing drainage.
- Conducting vehicle and equipment repair work at an offsite location unless not feasible, such as due to vehicle breakdown.
- Storing materials in original manufacturer containers and under roof cover if possible.

- Maintaining orderly storage areas to facilitate inspection.
- Disposing construction wastes in covered dumpsters or recycling containers.

5.0 SPILL PREVENTION, CONTROL, AND COUNTERMEASURES

ARCADIS will designate an onsite representative responsible for compliance management, including implementing and maintaining the spill prevention, control, and countermeasures described in the following sections. All personnel will be briefed on proper equipment operation and inspection procedures to minimize accidental release of fuels or other liquids, as well as on proper procedures to be followed during refueling.

Potential pollutant sources which could be introduced into the air, water, or soil at the site during construction include: chemicals of potential concern present in soils or groundwater, solids/liquids from decontamination and washing activities, dust and sediment generated during drilling operations, contractor-generated wastes (e.g., rubbish, excess construction materials), and vehicle fluids from heavy equipment and personnel vehicles (e.g., fuel, waste oil, antifreeze, etc.).

5.1 Designated Areas

Storage, handling, and transfer of hazardous and potentially hazardous materials will occur in designated areas, including the following:

- Fueling Areas – Refueling of vehicles and/or large equipment will be limited to designated fueling areas that are located away from storm drains. Refueling operations will be supervised at all times and appropriate spill containment and spill response equipment will be available. Refueling of small equipment may occur at the point of use but only if proper spill containment equipment is readily available (e.g., absorbent materials).

- Material and Equipment Staging Areas – When not in use, vehicles and equipment will be parked in a designated equipment staging area. Any hazardous materials and investigation-derived waste will be stored in a designated material staging area that is at least 100 feet away from any surface waters.

- Decontamination Areas – Decontamination facilities for equipment and personnel will be located in a designated decontamination area. Vehicles and equipment will be decontaminated before leaving the site as required.

5.2 Spill Response

Initial action and spill control are crucial for effective spill response. Following a spill, the initial actions include verifying the safety of nearby workers and the public (as necessary) and notifying designated spill response/emergency response personnel. During the control phase, responders organize the scene and mobilize equipment to control hazards and ensure effective cleanup. Spill response procedures are detailed in the project-specific Accident Prevention Plan/Site Safety and Health Plan (APP/SSHP ARCADIS 2013b).

5.3 Notification and Reporting

In the event of a spill, the Caretaker Site Office, Resident Officer in Charge of Construction, and the Navy RPM will be contacted. Contact information is provided below.

Caretaker Site Office	Melecio Asuncion	415-743-4721
Resident Officer in Charge of Construction	Izzat Amadea	510-749-5945
Navy RPM	Christopher Dirscherl	619-532-0785

5.3.1 Federal Requirements

ARCADIS is required to report any spill of oil, petroleum product, hazardous substance, or hazardous waste that meets the following criteria:

- Violates water quality standards
- Produces a visible sheen on surface waters
- Causes a sludge or emulsion to form on the water or exceeds reportable quantity

If one or more of these requirements are met, the National Response Center will be notified immediately (800-424-8802). The EPA Regional Administrator must be notified within 15 days of: a spill or release that requires activation of the spill controls and countermeasures; or a fire, explosion, or spill of any amount that reaches United States navigable waters.

5.3.2 California State Requirements

The State Office of Emergency Services must be notified of: any release of oil or hazardous material; or a spill or release of any material that may impose an environmental risk.

5.3.3 Local Requirements

If a release or spill of oil or gasoline poses a fire or explosion hazard, the local emergency response number should be called (911).

5.3.4 External Notification List

Agency and Responsibility Phone Numbers:

- National Response Center (800-424-8802)—Report spills to water that violate water quality criteria, cause a sheen, or exceed the reportable quantity
- State Office of Emergency Services Warning Control Center (800-852-7550)—Report oil or hazardous materials spills to land or water

- Local Fire and Emergency Services (911 or Vallejo Fire Department at 707-552-3285)—Report spills with fire or explosion potential

In regards to any reportable releases of oil, petroleum products, hazardous chemicals, or hazardous waste, an 'Emergency Release Follow-up Notice Reporting Form' from the California Environmental Protection Agency and Office of Emergency Services must be filled out.

6.0 REFERENCES

ARCADIS U.S., Inc. (ARCADIS). 2013a. Final Remedial Investigation Work Plan for the Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California. November.

———. 2013b. Accident Prevention Plan/Site Safety and Health Plan, Remedial Investigation for the Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California. November.

California Department of Toxic Substances Control (DTSC). 2009. Letter from Janet Naito to Michael Bloom (Navy). October 5.

City of Vallejo. 2007. Mare Island Specific Plan Amended June 2008.

Tetra Tech EM, Inc. (TtEM). 2000. Internal Draft Investigation Area H2 Remedial Investigation, Mare Island, Vallejo, California. December 1.

Trevet. 2012. Preliminary Assessment/Site Inspection (PA/SI) Work Plan, Defense Reutilization and Marketing Office South and Crate Test Area North, Mare Island Naval Shipyard, Vallejo, California. August.

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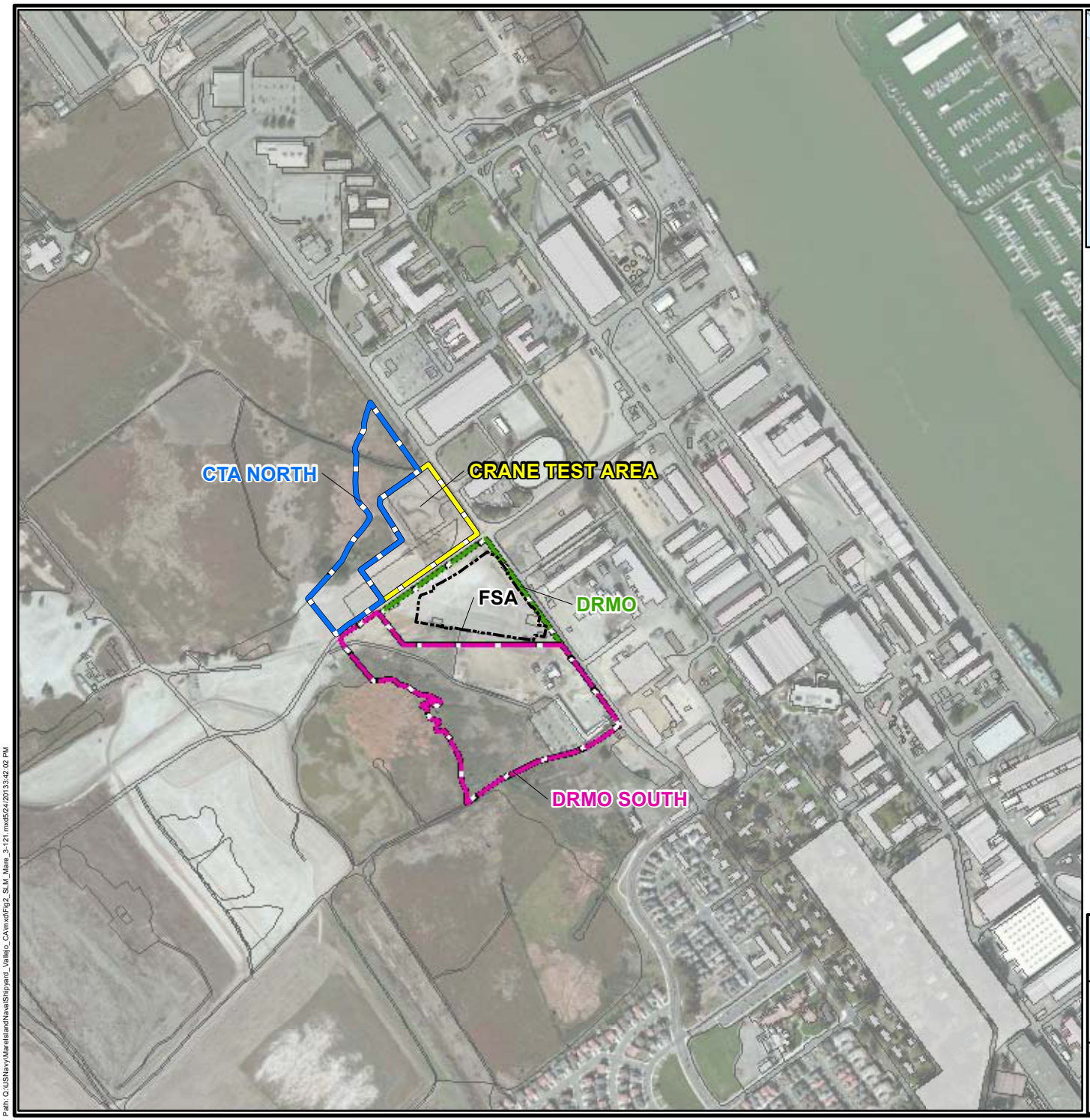

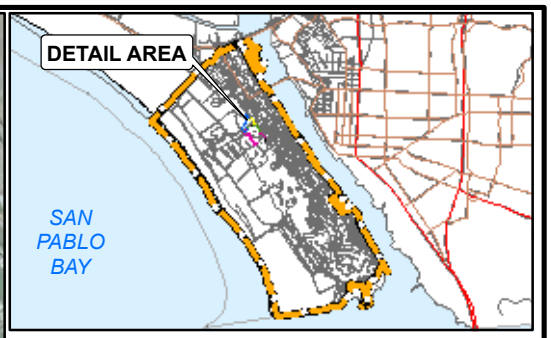

LEGEND:

- DRMO BOUNDARY
- DRMO SOUTH SITE BOUNDARY
- CTA NORTH SITE BOUNDARY
- CTA BOUNDARY (LENNAR MARE ISLAND)
- NAVY HISTORICAL BUILDING

ACRONYMS:
CTA - CRANE TEST AREA
DRMO - DEFENSE REUTILIZATION AND MARKETING OFFICE
FSA - FENCED SCRAPYARD AREA

SOURCE:
ENVIRONMENTAL SYSTEMS RESEARCH INSTITUTE (ESRI) USA SPATIAL FEATURE CLASSES. NAD83 STATE PLANE COORDINATE SYSTEM ZONE VI (FEET)
BASE MAP WAS OBTAINED FROM TREVET INC.
IMAGE © 2010 MICROSOFT CORPORATION AND ITS DATA SUPPLIERS, ACCESSED VIA ESRI ONLINE ON 3/26/13.

0 800 1,600
SCALE IN FEET

MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

SITE LOCATION MAP

ARCADIS

FIGURE
1

CITY: SAN FRANCISCO DIV/GROUP: ENV/GIS DB: User: MEstifanos LD: PIC: PM: TM:
PROJECT: FEASWT-004-0000
Path: Q:\US\navy\Mare\Island\MareIslandShipyard_Vallejo_CA\mxd\Fig2_SLM_Mare_3-121.mxd5/24/2013 3:42:02 PM

Appendix D: Responses to Agency Comments

DRAFT RESPONSE TO COMMENTS – Draft Remedial Investigation (RI) Work Plan for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area North (CTA), Former Mare Island Naval Shipyard, Vallejo, California, July 2013

CONTRACT: Contract Number N62473-11-D-2226, Task Order 0004

COMMENTS BY: California Department of Fish and Wildlife, Ms. Tami Nakahara	**REVIEW DATE:** Received September 23, 2013

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
1a	Page 5-3, Section 5.1 Biological, Avoidance, Minimization, and Mitigation Measures; and Appendix C: Environmental Protection Plan, Page 2-3, Section 2.3 Fish and Wildlife/Threatened, Endangered, and Sensitive Species 1st paragraph	The text states, "*There are no plans to collect samples from within 50 feet of the non-tidal wetlands as part of the RI*." However, on Figure 3 "DRMO South Proposed RI Sampling Locations", proposed soil sampling location DRMOS-SB30 appears to be within 50 feet of the non-tidal wetland. Please explain this discrepancy.	Proposed soil sampling location DRMOS-SB30 is not intended to be collected within the 50-foot buffer zone for the non-tidal wetland. To ensure that this is the case, the biological monitor will accompany the project team on the field visit to identify and mark/stake proposed sample locations. If the biological monitor determines that the proposed location of DRMOS-SB30 is within 50 feet of the non-tidal wetland, the location will be shifted such that it is no longer within the buffer zone.
1b	1st bullet	In the unlikely event that work is required within 50 feet of the non-tidal wetlands, the text states, "*A silt fence will be installed between the work sites and areas of non-tidal wetlands immediately following vegetation removal and before sampling activities begin to prevent entry of salt marsh harvest mice into cleared areas*." Please revise the text to state, "*If heavy equipment will be used within 50 feet of the non-tidal wetlands, a silt fence will be installed between the work sites and areas of non-tidal wetlands…*"	The text will be revised as recommended.

DRAFT RESPONSE TO COMMENTS – Draft Remedial Investigation (RI) Work Plan for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area North (CTA), Former Mare Island Naval Shipyard, Vallejo, California, July 2013

CONTRACT: Contract Number N62473-11-D-2226, Task Order 0004

COMMENTS BY: California Department of Fish and Wildlife, Ms. Tami Nakahara	**REVIEW DATE:** Received September 23, 2013

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
1c	3rd bullet	The text states, "*Each day, prior to the initiation of work within 50 feet of non-tidal wetlands, the biological monitor will thoroughly inspect the work area and adjacent habitat areas to determine if salt marsh harvest mice or rails are present in these areas and will remain on site throughout the day while work activities occur in these areas*." Please revise the text to state, "*…the biological monitor will thoroughly inspect the work area and adjacent habitat areas to determine if salt marsh harvest mice, rails, rare plants, and birds protected by the Migratory Bird Treaty Act are present in these areas…*"	The text will be revised as recommended.
2	Page 5-4, Section 5.1 Biological, Avoidance, Minimization, and Mitigation Measures; and Appendix C: Environmental Protection Plan, page 2-4, Section 2.3 Fish and Wildlife/Threatened, Endangered, and Sensitive Species	The text states, "*A biological monitor will be present during vegetation removal conducted within 50 feet of the non-tidal wetlands but not during work conducted in the upland areas outside of the 50-foot buffer zone*." Please revise the text to state, "*A biological monitor will be present during vegetation removal conducted within 50 feet of the non-tidal wetlands*." Please note, the biological monitor will need to be present during work conducted in the upland areas outside the 50-foot buffer zone when rare plants and nesting birds are present in these areas, in accordance with the biological avoidance and minimization measures listed on pages 5-1 to 5-2 and Appendix C, pages 2-1 to 2-3. The biological monitor will also need to be present on site to ensure compliance with all the appropriate biological avoidance, minimization, and mitigation measures listed in the work plan, for upland habitat areas as well as non-tidal	The text will be revised as recommended. The Navy recognizes that the presence of a biological monitor may also be required during work conducted in the upland areas outside the 50-foot buffer zone per Section 5.1, Bullet #4 (Page 5-2) and Bullet #5 (Page 5-2) of the Draft RI Work Plan (also Section 2.3, Bullets #4 and 5, Page 2-2 of Appendix C). The biological monitor will be responsible for ensuring compliance with the appropriate biological avoidance, minimization, and mitigation measures listed in the RI Work Plan.
2			

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
(continued)		wetland areas (i.e., confining heavy equipment to access routes, designated staging areas, and designated drilling areas). The biological monitor is responsible for preventing take of special status species and ensuring compliance with Applicable or Relevant and Appropriate Requirements.	
3	Appendix A: Sampling and Analysis Plan, page 51, Section 11.4.1 Lateral Boundaries	The text states, "*In addition, no samples will be collected from within 50 feet of the nontidal wetlands*." Please see Specific Comment 1a.	See response to Specific Comment #1a.
4	Appendix C: Environmental Protection Plan, page 2-1, Section 2.3 Fish and Wildlife/Threatened, Endangered, and Sensitive Species	The text states, "*Monitoring for the California clapper rail will be conducted in conjunction with salt marsh harvest mouse monitoring even though the California clapper rail is less likely to be encountered at DRMO South and CTA North*." Please revise the text to state, "*Monitoring for the California Clapper Rail and California Black Rail will be conducted in conjunction with salt marsh harvest mouse monitoring even though the California Clapper Rail and California Black Rail are less likely to be encountered at DRMO South and CTA North*."	The text will be revised as recommended.

References:

ARCADIS U.S., Inc. 2013. Draft Remedial Investigation Work Plan for the Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California. July.

DRAFT RESPONSE TO COMMENTS – Draft Remedial Investigation (RI) Work Plan for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North, Former Mare Island Naval Shipyard, Vallejo, California, July 2013

CONTRACT: Contract Number N62473-11-D-2226, Task Order 0004

COMMENTS BY: Department of Toxic Substances Control (Janet Naito) and the Regional Water Quality Control Board (Elizabeth Wells)	**REVIEW DATE:** Received September 23, 2013

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
General Comments			
1	General	Confirmation samples for the DRMO petroleum corrective action excavation were analyzed for total petroleum hydrocarbons (TPH) as Bunker C (C12-C40). The Draft RI Work Plan proposes analysis of soil and groundwater samples for TPH as diesel and as motor oil. Confirm that these analyses cover the same carbon range as the confirmation sampling previously conducted for Bunker C (C12-C40). If they do not, modify the analytical testing to include Bunker C range as well as diesel and motor oil.	The following hydrocarbon ranges will be specified for the analysis of total petroleum hydrocarbons (TPH) as diesel and motor oil, respectively: C10 to C28 for TPH as diesel, and C28 to C40 for TPH as motor oil. Thus, these analyses will cover the carbon range analyzed for (C12 to C40) during previous confirmation sampling conducted for Bunker C.
2	General	Replace "RWQCB" with "Water Board" throughout the document.	The global change will be made to replace "RWQCB" with "Water Board".
3	General	Screening Levels. DTSC recommends the use of the 2013 U.S. Environmental Protection Agency Regional Screening Levels (RSLs) in conjunction with DTSC's Human Health Risk Assessment (HHRA) Note 3 and Table 1. When referencing the RSLs, please reference the 2013 RSLs instead of the 2012 RSLs. When referencing CHHSLs, please reference the 2010 California Human Health Screening Levels (CHHSLs) instead of 2005 CHHSLs. The text and tables should be updated to reflect this. DTSC's Human Health Risk Assessment (HHRA) Note 3 and Table 1 can be found at: http://www.dtsc.ca.gov/AssessingRisk/upload/HHRA-Note-3-2.pdf.	Noted, applicable text and tables will be updated to reference the most recent versions of the screening criteria: i.e., the 2013 United States Environmental Protection Agency Regional Screening Levels (updated in May) and the 2010 California Human Health Screening Levels.

DRAFT RESPONSE TO COMMENTS – Draft Remedial Investigation (RI) Work Plan for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North, Former Mare Island Naval Shipyard, Vallejo, California, July 2013

CONTRACT: Contract Number N62473-11-D-2226, Task Order 0004

COMMENTS BY: Department of Toxic Substances Control (Janet Naito) and the Regional Water Quality Control Board (Elizabeth Wells)	**REVIEW DATE:** Received September 23, 2013

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
Specific Comments			
1	Section 1.3.3	Clarify the hydrogeology beneath the DRMO South and CTA North. The Draft RI WP states that a "single, shallow, unconfined water-bearing zone," extending to 28 feet below ground surface, is located beneath the adjacent DRMO. The Navy's June 3, 2013, request for a beneficial use exception describes two distinct hydrogeologic units beneath the DRMO South and CTA North. The Navy has identified an intermediate water-bearing zone at other locations on Mare Island; confirm whether this zone is absent beneath the DRMO South and CTA North.	Section 1.3.3 will be revised with the following language: "Three water-bearing zones (shallow, intermediate, and deep) have been identified at Mare Island. The shallow water-bearing zone refers to both saturated artificial fill and naturally deposited materials. In most areas of Mare Island outside the original island footprint (including the DRMO, DRMO South, and the CTA North), the shallow water-bearing zone is about 5 to 13 feet thick. Groundwater flow in the shallow water-bearing zone is complex and variable because of the heterogeneous nature of the artificial fill material on the island. However, groundwater flow direction generally varies from west to northwest at all three sites. The intermediate and deep water-bearing zones have not been observed in previous borings advanced at DRMO South and CTA North"
2	Section 2.3.1	Provide more details about the tank that was removed, such as identification number (if any), location (inside or outside building), containment structure, closure status, etc.	The text in Section 2.3.1 has been revised to the following: "A 500-gallon aboveground storage tank located outside of the building perimeter on the northwest side of Building 965 supported an emergency generator. As noted in the Mare Island Health, Safety, and Environmental Building Survey Building 965, dated October 30, 1996, the out-of-service tank was drained of diesel fuel, triple-rinsed, and sent for scrap off site. No containment area related to this tank is present, and there are no visible signs of its presence or past releases. Soil boring DRMOS-SB12 was installed in the area of the former tank to characterize the location."

DRAFT RESPONSE TO COMMENTS – Draft Remedial Investigation (RI) Work Plan for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North, Former Mare Island Naval Shipyard, Vallejo, California, July 2013

CONTRACT: Contract Number N62473-11-D-2226, Task Order 0004

COMMENTS BY: Department of Toxic Substances Control (Janet Naito) and the Regional Water Quality Control Board (Elizabeth Wells)

REVIEW DATE: Received September 23, 2013

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
3	Sections 3.1.1 and 3.1.2	Include in the discussion how the TPH as diesel and as motor oil relate to the TPH as Bunker C that was detected in the excavation confirmation samples.	The following text will be added to Sections 3.1.1 and 3.1.2: "Diesel generally consists of hydrocarbons in the carbon range of C10 to C28, while motor oil varies and can include hydrocarbons with carbon numbers ranging from 18 (C18) to greater than 36 (C36+). In comparison, the product targeted during the 2009-2010 Petroleum Corrective Action (PCA) demonstrated a hydrocarbon pattern similar to that of Bunker C fuel oil (i.e., C12 to C40), i.e., samples contained both diesel-range hydrocarbons and 'heavier-end' hydrocarbons in the motor oil range. Note that the following hydrocarbon ranges will be specified for the analysis of TPH-d and TPH-mo, respectively, as part of the RI: C10 to C28 for TPH-d, and C28 to C40 for TPH-mo. Thus, these analyses will cover the carbon range analyzed for (C12 to C40) during previous confirmation sampling conducted for Bunker C."
4a	Section 4.1, Table 4.1, Figure 3	Regional Water Board staff focused on the sampling locations proposed for further characterization of petroleum hydrocarbons and concur with many of the proposed locations. Comments about specific borings/samples are presented below: a. While proposed boring DRMOS-SB027 will assess vertical extent near the northern petroleum cleanup action (PCA) excavation boundary, no shallower sampling (less than 10 feet bgs) is proposed. Therefore, the lateral extent will not be defined in this area. Because no data is shown on Figure 3 (Proposed Sampling Locations), Regional Water Board staff reviewed Figure 11 of the Draft DRMO South PA/SI. It appears that data, and possibly boring numbers, are posted incorrectly so it is	The location of proposed boring DRMOS-SB27 was selected considering the historical detection of elevated TPH-d concentrations (i.e., 3,870 milligrams per kilogram [mg/kg]) in the soil sample collected from 10.5 feet bgs at 02GB026. TPH-d was not detected above analytical reporting limits in soil samples collected from shallower depths (less than 10 feet bgs) at 02GB026, i.e., 1.5 feet bgs and 7.5 feet bgs. Data for soil samples collected from less than 10 feet bgs is also available from the 2009-2010 PCA, which included the collection of confirmation sidewall samples from depths of less than 10 feet bgs along the excavation area's southeastern boundary. For example, sidewall confirmation sample F13SWSE, collected from 7.5 feet bgs near the location of 02GB026, had a TPH as Bunker C concentration of 100 mg/kg, below the Tier 2

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
4a (continued)		difficult to discern at what depths samples should be taken in this area.	Screening Level. Based on these historical results, the lateral extent of petroleum contamination in the area of DRMOS-SB27 has been effectually defined for soil at depths of less than 10 feet bgs. Note that the chemical data boxes for IR10GB050 and IR02GB026 were switched in Figure 11 of the Draft DRMO South PA/SI. With this correction, historical chemical results for IR02GB026 are consistent with the above discussion. A revised Figure 11 will be prepared.
4b	Section 4.1, Table 4.1, Figure 3	b. Show historical sample location DRMOGB046 on the Figure. Because this location is not shown on Figure 11 of DRMO South PA/SI, it is difficult to assess the proposed boring (DRMOS-SB34) near this location.	Historical sample location DRMOGB046 is shown on Figure 3 by the square marker located immediately south of proposed boring DRMOS-SB34. For clarification, sample identification labels will be added to Figures 3 and 4 (see attached) for the "previously collected" (i.e., pre-PA/SI) sample locations that are specifically called out in the text.
5	Table 4.1 and Section 4.1	Clearly state the rationale for the proposed groundwater sampling locations.	The following text will be added to Section 4.1: "Groundwater samples were proposed at boring locations DRMOS-SB36, DRMOS-SB37, and DRMOS-SB39 to assess potential impacts associated with groundwater migration from hydraulically upgradient source areas (e.g., IR10/SWMU 107 [the former PCB storage area] and IR14 [Pump Station 1 area]). A groundwater sample was proposed at boring location DRMOS-SB42 because groundwater data were not collected from the open space/undeveloped portion of the site during the 2012 site inspection." This rationale will be clearly stated in Table 4.1.

DRAFT RESPONSE TO COMMENTS – Draft Remedial Investigation (RI) Work Plan for the Defense Reutilization and Marketing Office (DRMO) South and Crane Test Area (CTA) North, Former Mare Island Naval Shipyard, Vallejo, California, July 2013

CONTRACT: Contract Number N62473-11-D-2226, Task Order 0004

COMMENTS BY: Department of Toxic Substances Control (Janet Naito) and the Regional Water Quality Control Board (Elizabeth Wells)

REVIEW DATE: Received September 23, 2013

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
6	Sections 4.2 and 5.4.1	Provide the rationale for using silica gel cleanup with the petroleum hydrocarbon analysis for soil samples. If previous testing from the petroleum excavations and PA/SI work was not performed using silica gel cleanup, discuss how the data will be directly comparable. Note that silica gel cleanup is proposed only for soil samples.	The silica gel cleanup method is used to isolate the non-polar carbon-hydrogen combinations that are associated with diesel and motor oil from the polar carbon-hydrogen combinations associated with the breakdown of organic matter. This step is valuable for sites, such as DRMO South and CTA North, with relatively high organic material content in soil. Note that silica gel cleanup was used for the confirmation soil samples collected during the 2009-2010 Petroleum Corrective Action as well as during the PA/SI work.
7	Section 5.4.2	Include how groundwater sampling locations will be monitored for separate phase product and actions to be taken if it is observed.	The last sentence of Page 5-7, 2nd Paragraph, will be modified as follows: "Field staff will also measure the groundwater level at each monitoring well using an electronic oil-water interface probe. If the probe indicates that separate phase product is present in the well, the thickness of the product will be measured and recorded on the groundwater sampling sheet. Separate phase product observed in the well will be removed to the extent possible during purging activities and prior to sample collection, and the groundwater sample will be collected from the approximate midpoint of the water column. If separate phase product cannot be removed during purging activities, a groundwater sample will not be collected." The same language will be added to Section 14.4 of SAP Worksheet #14.
8a	Sampling and Analysis Plan, Worksheet #9, Agenda Topic 2,	It appears some text is missing in the discussion.	The reviewer is correct. Under 'Discussion', the second sentence should read: "Chris provided an update on the PA/SI field work currently underway: currently conducting Geoprobe work in DRMO

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
8a (continued)	p. 30		proper, will move to DRMO-South this week and to CTA-North next week, wrapping up by October 12)." The missing text will be added to SAP Worksheet #9, Agenda Topic 2.
8b	Sampling and Analysis Plan, Worksheet #11, Section 11.1	The text does not include information about the elevated concentrations of Bunker C; see General Comment 1, above.	Bullet #1 (Page 49) of Worksheet #11, Section 11.1 will be revised as follows: • To further evaluate and/or delineate potential chemicals of potential concern (COPCs) identified in soil and groundwater at DRMO South and CTA North during the site inspections (SIs) conducted in October and November of 2012 and during previous investigations. Note that one objective of the 2012 SIs, as stated in the Preliminary Assessment/Site Inspection Work Plan, was to evaluate the extent of elevated concentrations of petroleum hydrocarbons (i.e., as Bunker C fuel oil) in soil near the 2009-2010 Petroleum Corrective Action excavation area on the southern boundary of CTA North (Trevet 2012). Samples collected from three boreholes (CTAN-SB11, CTAN-SB12, and CTAN-SB13) advanced near the northern limit of the excavation satisfied this objective. These samples did not exhibit elevated petroleum hydrocarbon concentrations above the screening criteria (see Section 10.6.1.2). In addition, the below sentence will be added following Bullet

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
8b (continued)			#3 on Page 50 (Section 11.1 of Worksheet #11): "Note that the TPH-d and TPH-mo analyses are anticipated to cover the hydrocarbon range observed for the product that was targeted during the 2009-2010 PCA (i.e., C12 to C40, Bunker C fuel oil)."
8c	Sampling and Analysis Plan, Worksheet #14, Section 14.4	Include monitoring for separate phase product prior to pumping the wells. In addition, discuss how a representative sample of groundwater will be collected for analysis of dissolved petroleum constituents if product is present.	See also response to Specific Comment #7. SAP Worksheet #14, Section 14.4, 3rd Paragraph on Page 62 will be revised as follows: "…Field staff will also measure the groundwater level at each monitoring well using an electronic oil-water interface probe. If the probe indicates that separate phase product is present in the well, the thickness of the product will be measured and recorded on the groundwater sampling sheet. Depth to groundwater and, if applicable, separate phase product (to the nearest 0.01 foot) will be measured from the top of PVC casing (at the permanent mark/notch placed on the north side), and distance from ground surface to top of PVC casing will be measured and recorded in the boring log and field logbook. Separate phase product observed in the well will be removed to the extent possible during purging activities and prior to sample collection, and the groundwater sample will be collected from the approximate midpoint of the water column. If separate phase product cannot be removed during purging activities, a groundwater sample will not be collected."
8d	Sampling and Analysis Plan, Various (Worksheets	Please note that comments on the main text also apply to the applicable sections of the SAP (including, but not limited to Worksheets #10, #14, and #17).	Noted. Revisions to the RI Work Plan (i.e., in response to Specific Comment #3, #4b, #5, and #7) will also be incorporated into the applicable sections of the SAP.

NO.	REFERENCE	REVIEW COMMENT	RESPONSE
8d (continued)	#10, #14, #17)		

References:

ARCADIS U.S., Inc. 2013. Draft Remedial Investigation Work Plan for the Defense Reutilization and Marketing Office South and Crane Test Area North, Former Mare Island Naval Shipyard, Vallejo, California. July.

Tetra Tech EM, Inc. (TtEM). 2000. Internal Draft Investigation Area H2 Remedial Investigation, Mare Island, Vallejo, California. December 1.

Trevet. 2012. Preliminary Assessment/Site Inspection (PA/SI) Work Plan, Defense Reutilization and Marketing Office South and Crane Test Area North, Mare Island Naval Shipyard, Vallejo, California. August.

Attachments:

Figure 3 – DRMO South Proposed RI Sampling Locations
Figure 4 – CTA North Proposed RI Sampling Locations