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Naval Facilities Engineering Command
Naval Surface Warfare Center
Crane, Indiana

Mine Fill A Battery Site Cleanup
Final Work Plan
NSWC Crane
Crane, Indiana

January 2001

TOLTEST, INC.

FINAL
INTERIM MEASURES WORK PLAN

MINE FILL A BATTERY SITE CLEANUP AT THE NAVAL SURFACE WARFARE CENTER CRANE, INDIANA

CONTRACT NO. N68950-96-D-0052
DELIVERY ORDER # FC04
TOLTEST PROJECT NO. 37826.01

JANUARY 2001

Submitted to:

OFFICER IN CHARGE OF NAVFAC CONTRACTS
NAVAL SURFACE WARFARE CENTER
BUILDING 2516
300 HIGHWAY 361
CRANE, INDIANA 47522-5082

Submitted by:

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NSWC CRANE

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SWMU 12/14

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LIST OF ACRONYMS

AHA	Activity Hazard Analysis
CFR	Code of Federal Regulations
COTR	Contracting Officers Technical Representative
CRZ	Contaminant Reduction Zone
DO	Delivery Order
DQOs	Data Quality Objectives
ECOTR	Environmental Contracting Officer Technical Representative
EE/CA	Engineering Evaluation/Cost Analysis
ES	Environmental Specialist
EZ	Exclusion Zone
FM	Facility Manager
FSP	Field Sampling Plan
HS	Hazardous Substance
MFA	Mine Fill A
MS/MSD	Matrix Spikes/Matrix Spike Duplicate
MSDS	Material Safety Data Sheets
NSWC	Naval Surface Warfare Center
OSHA	Occupational Safety and Health Administration
PCBs	Polychlorinated Biphenyl
PM	Project Manager
PPE	Personal Protective Equipment
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOPs	Standard Operating Procedures
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SVOCs	Semi-Volatile Organic Compound
SZ	Support Zone
TCLP	Toxicity Characteristic Leaching Procedure
USDOT	United States Department of Transportation
U.S.EPA Reg. V	United States Environmental Protection Agency, Region V
VOCs	Volatile Organic Compounds
WP	Work Plan

TABLE OF CONTENTS

Page No.

List of Acronyms iii

1.0 INTRODUCTION 1

1.1 Site Description 2
1.2 Site Physical Characteristics 3

2.0 CONSTRUCTION EQUIPMENT AND PERSONNEL 4

3.0 EXCAVATION SEQUENCE AND OPERATIONAL APPROACH 9

3.1 Permitting and Notification 9
3.2 Pre-Excavation Site Delineation and Sampling 9
3.3 Analytical Requirements 10
3.3.1 Soil Area 10
3.3.2 Battery Area 11
3.3.3 Background 11
3.4 Soil Excavation 12
3.5 Waste Management 13
3.6 Confirmatory Sampling 13
3.7 Site Restoration 14
3.8 Equipment and Personnel Decontamination 14
3.8.1 Equipment 14
3.8.2 Sampling Equipment 14
3.8.3 Personnel 15
3.9 Cleanup and Emergency Response 15

4.0 ENVIRONMENTAL PROTECTION 16

5.0 SITE SAFETY AND HEALTH 17

6.0 REPORTING REQUIREMENTS 18

7.0 SCHEDULE 19

TABLE OF CONTENTS (Continued)

FIGURES

Figure 1	NSWC Crane Vicinity Map
Figure 2	MFA Battery Area Map
Figure 3	Project Organizational Chart
Figure 4	Schedule Time Line

APPENDICES

APPENDIX A	Site Safety and Health Plan
APPENDIX B	Sampling and Analysis Plan
APPENDIX C	Quality Assurance Project Plan
APPENDIX D	Activity Hazard Analysis

1.0 INTRODUCTION

TOLTEST has been retained by the Naval Facilities Engineering Command Officer in Charge of Construction under contract N68950-96-D-0052, Deliver Order (DO) #FC04 to provide an Interim Measures Work Plan (WP), Site Safety and Health Plan (SSHP), Sampling and Analysis Plan (SAP), and Quality Assurance Project Plan (QAPP), for completing remediation activities at the Mine Fill A (MFA) Battery Disposal and Soils area (Battery Site), Naval Surface Warfare Center (NSWC) Crane, Indiana. This plan will be reviewed and approved by the Contracting Officers Technical Representative (COTR), Environmental Contracting Officer Technical Representative (ECOTR) and the United States Environmental Protection Agency Region V (U.S.EPA Reg. V), prior to the start of any work.

The objective of this work is to characterize the area via soil analysis, remove battery fragments and potentially contaminated soil from the impacted areas, properly dispose of contaminated material, complete post-excavation confirmatory sampling and analysis, implement site restoration to prevent excessive soil erosion, and complete documentation and reporting to the ECOTR. Since this is an interim measure, this scope of work addresses only the removal of the contamination source and does not address the potential for groundwater contamination.

This WP includes the sequencing of all remediation activities and procedures to be followed during all phases of the remediation. This WP is supplemented by the SSHP (Appendix A), the SAP (Appendix B), and the QAPP, (Appendix C). The WP and SSHP conform to Occupational Safety and Health Administration (OSHA) regulations identified in 29 Code of Federal Regulations (CFR) 1910.120 "Hazardous Waste Operations and Emergency Response" and applicable parts of 29 CFR 1920 and 1926. A copy of this WP will be on site during remedial activities.

Remediation activities will be conducted in accordance with U.S.EPA Reg. V, Indiana Department of Environmental Management, and United States Department of Transportation (USDOT 49CFR) regulations.

1.1 Site Description

The NSWC at Crane is located in southwestern Indiana approximately 30 miles southwest of Bloomington and occupies the northern half of Martin County and parts of Lawrence, Greene, and Davies counties (illustrated on Figure 1). The NSWC provides support for equipment, shipboard weapons systems, and ordnance. In addition, the NSWC supports the Crane Army Ammunition Activity including production and renovation of conventional ammunition, storage, shipment, and demilitarization and disposal of conventional ammunition.

The Battery Site is located on the south end of MFA approximately 140 feet outside the perimeter fence, accessed through a gate (see Figure 2). The site consists of two areas: 1) where batteries were dumped on the ground surface (referred to as the battery area), and 2) an adjacent area where soil and construction debris was dumped (referred to as the soil area). The origin of these areas is unknown: the history of when they were placed, by whom, or for what reason is not available. It is a reasonable assumption that the mounded soil in the soil area originated from within MFA.

The site was discovered early in the 1990s when a representative of the Crane Environmental Protection Department (EPD) noticed the mounded soil and became suspicious. Further investigation revealed the presence of the battery disposal area. At that time, the batteries were recognizable as AA household type and their numbers were estimated to be in the thousands.

At present, the batteries are all but unrecognizable and only their inner core is visible on the ground surface. The soil area has no apparent unusual characteristics: no staining is present and no odor is detectable. The size of the soil area is approximately 2355 square feet consisting of several soil mounds, none exceeding four feet in height. The size of the battery area is approximately 3500 square feet. The potential for the waste from these areas to be a listed or characteristic waste is unknown.

1.2 Site Physical Characteristics

The soil area resides in a flat and level grassy area, and large pieces of concrete can be found around the perimeter with smaller pieces of concrete and drainage tile mixed in with the soil. The battery area is approximately four feet lower in elevation than the soil area. The battery area is flat but slopes slightly to the southwest in the direction of surface drainage (see Figure 2).

Surface soil types at the site are described in the Martin County Soil Survey [1] as a Zanesville silt loam, moderately sloping, and well to moderately well drained. A fragipan is typically present at a depth of 23 to 31 inches. Permeability is moderate above the fragipan and slow in the fragipan. According to Halliburton [2] no specific information on the hydrogeology or bedrock geology of the MFA area is available.

2.0 CONSTRUCTION EQUIPMENT AND PERSONNEL

Table 1.0 lists the equipment expected to be used and TOLTEST personnel positions assigned to this cleanup activity.

TABLE 1.0	
Equipment	**TolTest Key Personnel**
Backhoe/Excavator Rubber Tire Loader Dump Trucks Assorted hand tools Personal Protective Equipment Decontamination Equipment Sampling Equipment	NSWC Facility Manager Project Manager QA/QC Representative Health and Safety Officer Site Supervisor Environmental Specialist Environmental Technician Operators/Drivers Laborer

Figure 3 provides a Project Organizational Chart illustrating how TOLTEST relates to the regulating agencies involved in this project. The agencies involved and TOLTEST'S personnel positions are described below.

NAVFACENGCOM Southern Division (South Div)
Overview of project execution and coordination between TOLTEST, NSWC Crane, US EPA, and other agencies.

US EPA Region V
Responsibilities include overview of all site activities to assure compliance with RCRA, and approval of all work plans and reports.

NSWC Crane Officer In Charge of Construction (OICC)
South Div's on-site representative and liaison between NSWC Crane, US EPA, and TOLTEST.

NSWC Crane Environmental Protection Department (EPD)

The EPD is responsible for monitoring TOLTEST's performance for compliance with RCRA and other pertinent environmental regulations. A representative from EPD will sign, as the generator, any manifests for waste materials requiring disposal.

TOLTEST Facility Manager

TOLTEST's Facility Manager (FM) is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The FM will report directly to the ECOTR and will provide the major point of contact and control for matters concerning the project.

The FM will approve all reports (deliverables) before their submission to the U.S. Navy and U.S.EPA, Reg. V, and ultimately be responsible for the preparation and quality of interim and final closure reports.

TOLTEST Project Manager

TOLTEST's Project Manager's (PM) primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The PM has responsibility for ensuring that the project meets objectives and TOLTEST's quality standards. The PM will:

- Develop a detailed work plan schedule;
- Establish project policy and procedures to address the specific needs of the project as a whole, and the objectives of each task;
- Orient the Site Supervisor and all support staff concerning the project's special considerations;
- Monitor and direct the Site Supervisor;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements and authorizations.

Quality Assurance/Quality Control (QA/QC) Representative

The qualifications and requirements of the QA/QC Representative are in accordance with the Department of the Navy, Naval Facilities Engineering Command, Guide Specification NFGS-01450J dated 31 March 2000.

The QA/QC Representative will implement and manage the QC program on this project. This individual will remain independent of direct job involvement and day-to-day operations, and will have direct access to corporate executive staff as necessary to resolve any QA dispute. He/she is responsible for auditing the implementation of the QA program in conformance with this plan and TOLTEST'S policies. Specific functions and duties include:

- Providing QA audits on various phases of the field operations;
- Reviewing and approving of QA plans and procedures;
- Providing QA technical assistance to project staff;
- Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the FM and PM.

Health and Safety Officer

The qualifications and requirements of the Health and Safety Officer are in accordance with the Department of the Navy, Naval Facilities Engineering Command, Guide Specification NFGS-01525F dated 31 March 2000.

The Health and Safety Officer (HSO) reports to the PM and implements and ensures compliance with the Site Safety and Health Plan (SSHP) and tracks and reports on safety related matters. Specific responsibilities include:

- Implements personnel surveillance program;
- Provides site-specific training as required by the SSHP;
- Conducts audits as appropriate to ensure compliance;
- Stops work when necessary to ensure the safety of personnel and to prevent damage to the environment.

Site Supervisor

The TOLTEST Site Supervisor will be responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision and will report directly to the PM. Specific responsibilities include:

- Provision of day-to-day coordination with the PM on technical issues in specific areas of expertise;
- Implementing field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements;
- Coordinating and managing field staff activities including sampling, excavating, and site restoration;
- Adhering to work schedules;
- Coordinating and overseeing of technical efforts of subcontractors assisting the field team;
- Identifying problems at the field team level, resolving difficulties in consultation with the PM, implementing and documenting corrective action procedures and provisions of communication between team and upper management;
- Documenting daily field activities on the Contractors Production Report and submitting the report on a daily basis to the ECOTR.
- Participating in preparation of the Closure Report.

Environmental Specialist

The Environmental Specialist (ES) will be responsible for implementing the requirements of the SAP and QAPP in the field to ensure that project objectives are met. Duties will include supervision of grid layout in the contaminated areas, coordination of pre and post-excavation soil sampling, and proper shipment of samples to the laboratory. The ES will interpret and report results of analytical data to the ECOTR, FM, and PM. The ES will coordinate the proper disposal of contaminated soils and debris removed from the site in conjunction with the ECOTR.

Environmental Technician

The Environmental Technician (ET), under the supervision of the ES, will assist in grid layout in the contaminated areas, perform pre and post-excavation soil sampling activities, and prepare samples for shipment to the laboratory

Operators/Drivers

Equipment operators will be responsible for proper operation and maintenance of heavy equipment utilized on the site. They will be familiar with the scope of work and will be under the direct supervision of the Site Supervisor.

Laborer

Laborers will report to the Site Supervisor and will be responsible for general site cleanup and maintenance, hand digging, and decontamination of equipment.

3.0 EXCAVATION SEQUENCE AND OPERATIONAL APPROACH

The objective of this interim measure is to characterize the battery area and soil area via soil analysis, remove battery fragments and potentially contaminated soil from the impacted areas, properly dispose of contaminated material, complete post-excavation confirmatory sampling and analysis, implement site restoration to prevent excessive soil erosion, and complete documentation and reporting to the ECOTR. The excavation sequence and operational approach to complete these objectives are defined in the following sections.

3.1 Permitting and Notification

The NSWC Crane facility requires issuance of permits before initiation of excavation activities. TolTest's Project Manager will be responsible for obtaining an Excavation and Trenching Permit through the Officer in Charge of Construction, Public Works Department, Code 094, Building 2516. A Safety Permit is also required and will be secured from the Crane Army Ammunition Activity located in Building 13. An unexploded ordnance clearance will be obtained, if necessary, from the ECOTR.

A Pre-Construction meeting will be conducted on the site between the NSWC Crane and TolTest prior to the start of any work. At this time, details of the sequencing of work, quality control requirements, and site health and safety procedures will be reviewed.

3.2 Pre-Excavation Site Delineation and Sampling

Once the WP, SSHP, SAP, and QAPP have been approved, TOLTEST will perform pre-excavation sampling activities. Representatives of TOLTEST, EPA, and EPD conducted a site walk of the site and delineated the boundaries of potential contamination. These boundaries were marked with pin flags at that time.

The number of pre-excavation sample points is based on the size of the areas to be sampled. A full discussion of the procedures for determining the number of sample points can be found in Section 2.3 of Appendix B. Following those procedures, a total of 11 pre-excavation grab samples will be obtained from the site: five from the soil area and six from the battery area.

The five samples from the soil area will be obtained from the soil mounds at varying depths from the surface to deep within the mounds. Samples from the battery area will be obtained from the surface to a depth of 12 inches. All samples will be grab samples, no composite samples will be obtained.

Sample points will be identified in the field with pin flags with the sample identifier written on the flag in indelible ink. The Pre-Excavation Sampling Location Map is depicted as Figure 1 in Appendix B.

3.3 Analytical Requirements

3.3.1 Soil Area

One sample from the soil area will be analyzed for explosives by EPA Method 8330 and Appendix IX[2] constituents consisting of metals, cyanide, sulfide, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), herbicides, and pesticides/PCBs. No analysis for dioxins/furans will be completed. The Appendix IX list can be found as Attachment 2 to Appendix B. Analysis for VOCs will conform to SW-846, method 8260/5035 for EnCore samplers. The remaining four samples from the soil area will be analyzed for Mine Fill A constituents (based on the Biofacility QAPP requirements): explosives, metals, and VOCs. These constituents were chosen based on the reasonable assumption that the soil in this area originated from Mine Fill A and the fact that, according to Crane EPD, there is no history of dioxin/furan contamination at Crane.

Soil that is shown to be contaminated with Mine Fill A constituents only will be handled and processed according to requirements set forth in the Biofacility Work Plan and QAPP. Crane EPD will be contacted for further direction if the soil is shown to be contaminated with Appendix IX constituents.

3.3.2 Battery Area

Pre-excavation samples from the battery area will be analyzed for toxic characteristics and metals as listed in Table 1.0.

Table 1.0
Battery Area Constituent List

CONSTITUENT	METHOD
Ignitability	1010
Corrosivity	EPA 150.1
Reactivity	Cpt. 7, SW 846
RCRA Metals	6010 & 7471
Cobalt	6010
Lithium	6010
Manganese	6010
Nickel	6010
Zinc	6010

The additional metals chosen (over and above the 8 RCRA metals) were based on constituents listed in several Material Data Safety Sheets (MSDSs) for batteries, and Appendix IX metals. Copies of the MSDSs are provided as Attachment 3 to Appendix B.

Analysis for disposal characterization (to include TCLP) will be completed if total metals analysis indicates that the contamination, if present, exceeds established cleanup goals.

Post-excavation samples will be analyzed for total metals only.

3.3.3 Background

No background samples will be obtained. The background levels for inorganic compounds as established in the Basewide Background Soils Study will be referenced as discussed in Section 2.5 of the SAP and Section 8.0 of the QAPP. Background levels for organic compounds are assumed to be zero.

3.4 Soil Excavation

Excavation will not be required if there are no contaminants present above established cleanup goals. Establishment of cleanup goals is discussed in Section 8.0 of the QAPP. Soil and debris from the Battery Area that are determined to be non-hazardous but above cleanup goals will be scheduled for excavation and disposal at a special-waste disposal facility. Soil and debris from the battery area that are determined to be hazardous will be scheduled for excavation and disposal at an approved hazardous waste disposal facility.

No soil excavation is expected in the Soil Area. Therefore, TOLTEST will notify the OICC and the ECOTR for further direction if contaminated soil is detected in the Soil Area which requires disposal.

The boundaries of soil excavation in the Battery Area will be based on analysis of pre-excavation samples. TOLTEST'S Environmental Specialist will confer with the ECOTR to delineate the boundaries of excavation. The depth of soil excavation will be approximately eighteen inches (since pre-excavation samples are obtained to a depth of one foot).

No contaminated soil will be excavated until the required disposal permits have been secured from the disposal facilities and transportation to the facilities has been arranged. The disposal facilities selected by TOLTEST will be approved by the ECOTR. The use of dump trucks or rolloff boxes for transportation will be arranged with the disposal facility.

Every effort will be made to excavate all contaminated soil in one day and avoid precipitation events. Open soil containers will be covered with plastic and open excavations will be lined with plastic if precipitation occurs or if excavation requires multiple days to complete. Straw bales will be placed around the excavation in an attempt to divert any runoff water from entering the excavation should precipitation occur. Any water collecting in the excavation will be pumped to containers and characterized for disposal purposes.

The areas of potential contamination are small enough such that the equipment used to excavate the contaminated soil will remain outside the limits of contamination, only the bucket will be in contact with the contaminated soil. The disposal containers (whether rolloff boxes or dump trucks) will be placed adjacent to the excavation areas so that the soil can be loaded directly into the containers. Excavating equipment and disposal

containers will be placed on plastic sheeting or felt during loading to prevent spillage from contacting uncontaminated soil.

In the event of precipitation, excavation activities will be stopped and the plastic sheeting or felt described above will be rolled up and placed inside a disposal container to prevent runoff from contaminating an otherwise uncontaminated area.

3.5 Waste Management

Liners will be placed inside the disposal containers prior to being loaded with contaminated soil. The containers will be tarped before being shipped to prevent spillage from the containers and infiltration of precipitation. Labels and placards supplied by the disposal facility, appropriate for the load being transported (either special or hazardous waste), will be affixed to each disposal container. Shipping manifests will be completed by TolTest and signed by the ECOTR as the generator. Shipping manifests will accompany all loads to the disposal facility. A copy of the manifests will be retained by the ECOTR and TOLTEST. Copies of all manifests and Certificates of Disposal/Recycling completed by the disposal facility will be returned to the ECOTR within 30 days of shipment off station.

3.6 Confirmatory Sampling

When all the identified contaminated areas have been excavated, post-excavation confirmatory samples will be collected to document removal of contamination. The number of samples to be obtained (both excavation floor and sidewall) will be based on the area of the excavation and length of the sidewalls as recommended in the Verification of Soil Remediation Guidance Document (Attachment 1of Appendix B). All samples will be grab samples obtained from the surface to six inches deep. Sample points will be identified in the field with pin flags with the sample identifier written on the flag in indelible ink. The excavations will be covered with 6-mil plastic sheeting following the completion of confirmatory sampling to prevent the accumulation of rainwater.

Confirmatory samples will be analyzed for those constituents detected in pre-excavation site characterization samples above cleanup goals. TolTest will contact the ECOTR and the OICC for further direction if analysis indicates that contamination above cleanup goals is still present on the floor or in the sidewalls of the excavation.

3.7 Site Restoration

Site restoration will consist of placing grass seed covered with straw on the excavation areas. The use of topsoil or backfill is not within the current scope of work for this project. Once the site restoration has been completed, TOLTEST will demobilize from the site.

3.8 Equipment and Personnel Decontamination

3.8.1 Equipment

Heavy equipment utilized for excavation of the contaminated soil will be decontaminated prior to leaving the site. Contamination of the equipment tracks or tires will be minimal since it will have been placed on a protective barrier during excavation. At the conclusion of excavation activities, contaminated soil adhering to the bucket of the equipment will be scraped off and loaded into the last remaining disposal container (dump truck or rolloff). To remove the remaining contaminated soil, the bucket will be held over a bermed decontamination pad constructed of 6-mil plastic sheeting. A hand held pump sprayer and brushes will be used to remove the remaining soil followed by a final fine mist rinse. A minimal amount of water will be used in this process. The soil and water that has collected in the decontamination pad, and the plastic from the pad and beneath the equipment and containers, will be placed in the disposal container. Shovels, scrapers, or other tools used to decontaminate the bucket will be cleaned in a similar manner.

3.8.2 Sampling Equipment

Any non-disposable sampling equipment and PPE will be decontaminated according to the following procedures;

- Wash in soapy water (alconox or equivalent)
- Rinse in potable water
- Rinse with 10% nitric acid
- Rinse with reagent grade water
- Air dry.

Decontamination wash water will be kept to less that two gallons per sampling event. All sampling equipment decontamination fluids will be collected, containerized, and characterized for disposal purposes. Analysis of decontamination water will be based on constituents that are detected in pre-excavation sampling and other analyses that may be required by the disposal facility (if disposal is off-site).

3.8.3 Personnel

Personnel are only expected to enter the work zone for sampling activities. Personnel protective equipment (PPE) consists of disposable clothing of Tyvek suits, over-boots, and nitrile gloves. Since this clothing is disposable, no decontamination water will be generated for personnel decontamination.

3.9 Cleanup and Emergency Response

Each day upon completion of work, TOLTEST will remove all excess materials and debris from the NSWC Crane property. The work areas will be left in a neat and clean condition and all materials and/or debris will be disposed of properly.

All hazardous substance (HS) releases or spills involving NSWC Crane waste or property will be immediately reported to the COTR and the ECOTR. Off-site HS releases will be immediately reported to the proper Federal, State, and Local agencies and the ECOTR. Spill cleanup, remediation and damage to the environment resulting from TOLTEST'S actions will be the responsibility of TOLTEST.

4.0 ENVIRONMENTAL PROTECTION

TOLTEST will provide and maintain environmental protection through the completion of this project. A pre-work meeting will be held prior to the commencement of remediation activities to discuss the proposed environmental protection. Environmental protection will be provided to correct conditions that develop during the project, or that are required to control pollution that develops during normal work practices. TOLTEST'S operations will comply with all Federal, State and Local regulations pertaining to water, air, solid waste, hazardous waste and substances, oily substances, and noise pollution.

TOLTEST will at all times be aware of and adhere to all applicable environmental laws, regulations, and environmental protection policies in effect at the NSWC Crane. Information concerning Navy environmental policies will be obtained from OPNAVINST 5090.1B and the NSWC Hazardous Waste Management Plan. TOLTEST will at all times comply with 40 CFR 262, 263, 264 and 265.

Any hazardous waste created, used, or brought onto NSWC Crane property by TOLTEST will be the responsibility of TOLTEST. Storage of hazardous waste will be in approved and properly labeled containers (49 CFR 178). The waste will be properly labeled with the proper shipping description, accumulation start date, and generator information. The containers (if any) will be removed from the project site, stored, and treated/disposed of in accordance with 40 CFR 263, 264.

All waste will be manifested from the site and will be handled through the NSWC Crane, ECOTR, who will approve and sign for all waste manifested from the site (as stated in section 3.5). Manifesting arrangements will be scheduled 24 hours in advance, excluding emergencies. After hours, weekend, and holiday, manifesting can be requested in writing 48 hours prior to shipment. NSWC Crane reserves the right to refuse all after hour, weekend, and holiday manifesting at the convenience of NSWC Crane.

TOLTEST will preserve the integrity of the natural resources of the project area. This includes returning the site to its approximate pre-construction conditions, ensuring that the surrounding area is not environmentally damaged in any way, and preventing the release of any hazardous substances into the surrounding air, land or water.

5.0 SITE SAFETY AND HEALTH

Site safety and health is described in detail in the SSHP, Appendix A. In addition, activity hazard analysis (AHAs) have been prepared for each definable feature of work. The SSHO will be responsible for reviewing the AHA with the all field personnel involved in a specific task. The following describes each task/operation in terms of the definable features associated with each major phase of work. AHAs have been prepared for each major phase of work and are presented in Appendix D. Definable features of work include the following:

a. Site Delineation and pre-excavation sampling
b. Soil Excavation and Removal
c. Disposal Activities
d. Post-excavation or Confirmatory Soil Sampling
e. Equipment Decontamination
f. Site Restoration

6.0 REPORTING REQUIREMENTS

TOLTEST will develop a detailed report containing all closure documentation including; tabulated analytical results for confirmatory samples; excavation of battery fragments and soil; transportation and disposal documentation; analytical results compared to regulatory limits; all original lab results; photographic documentation; copies of waste manifests; chain-of-custody documentation; and a discussion of all work performed. This report will be completed within 120 days after fieldwork is completed.

7.0 SCHEDULE

The work schedule will be eight to ten-hour days, five to six days per week. The construction sequence will begin once approvals and all necessary permits and notifications have been received. All fieldwork will be completed within six months after date of order to proceed by the government. The Schedule Time Line will be presented as Figure 4 after approval of this WP.

The work will be conducted in such a manner as to cause the least interference with the normal functions of the site and surrounding area. Portions of the site will be vacated for periods of time as necessary for TOLTEST to perform certain work, stage equipment and materials. Prior to beginning any work, TOLTEST will meet with the ECOTR in order to approve the site requirements, schedule, and sequence of work.

FIGURES

FIGURE 1 NSWC CRANE VICINITY MAP BATTERY SITE CLEAN UP NAVAL SURFACE WARFARE CENTER, MINE FILL A CRANE, INDIANA	DRAWN MRC\3-20-00	CHECKED
	REVISED	APPROVED
	JOB NO. 37826.01	TOLTEST, INC.
	DRAWING NUMBER 378265	

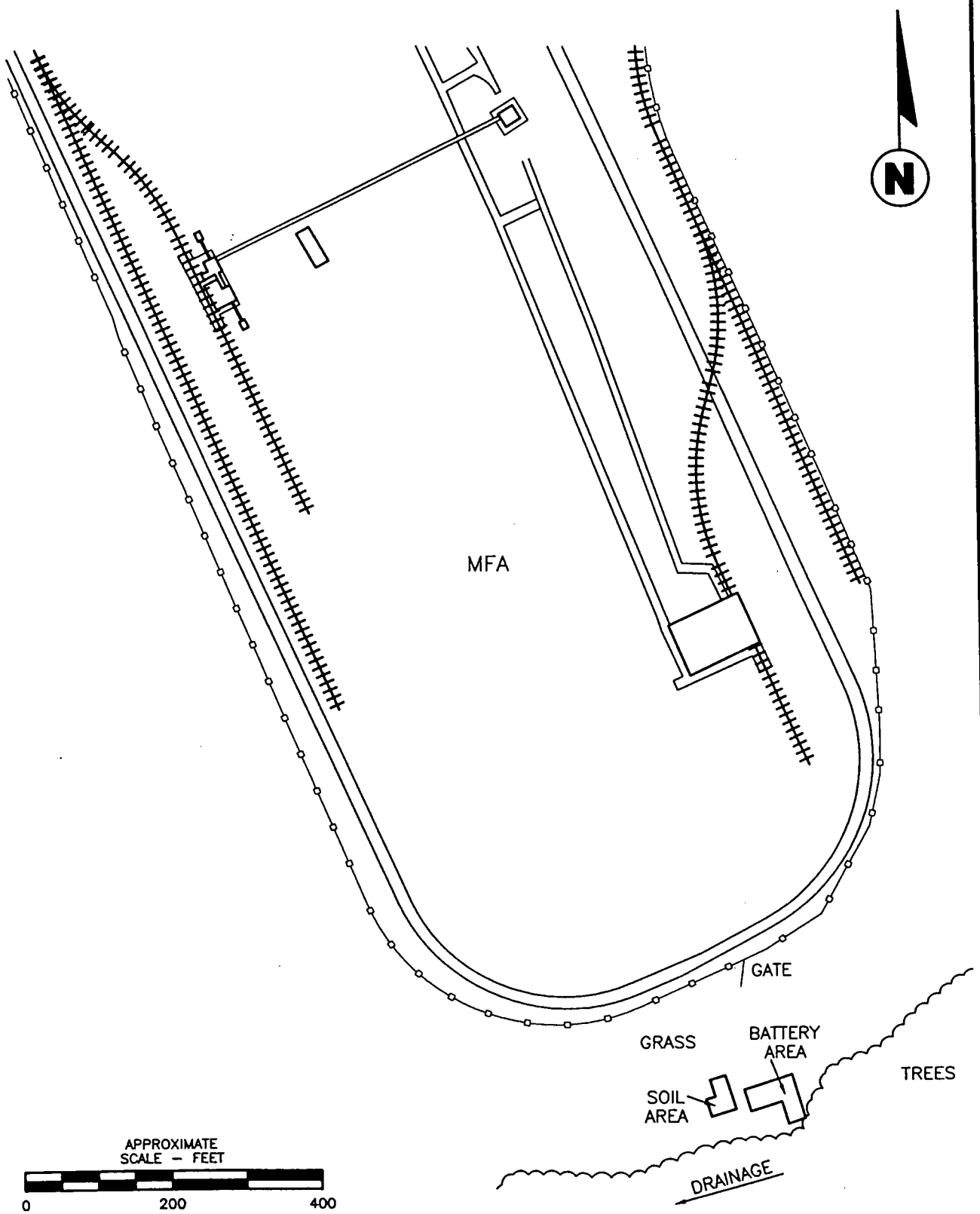

FIGURE 2

MFA BATTERY AREA MAP

BATTERY SITE CLEAN UP
NAVAL SURFACE WARFARE CENTER, MINE FILL A
CRANE, INDIANA

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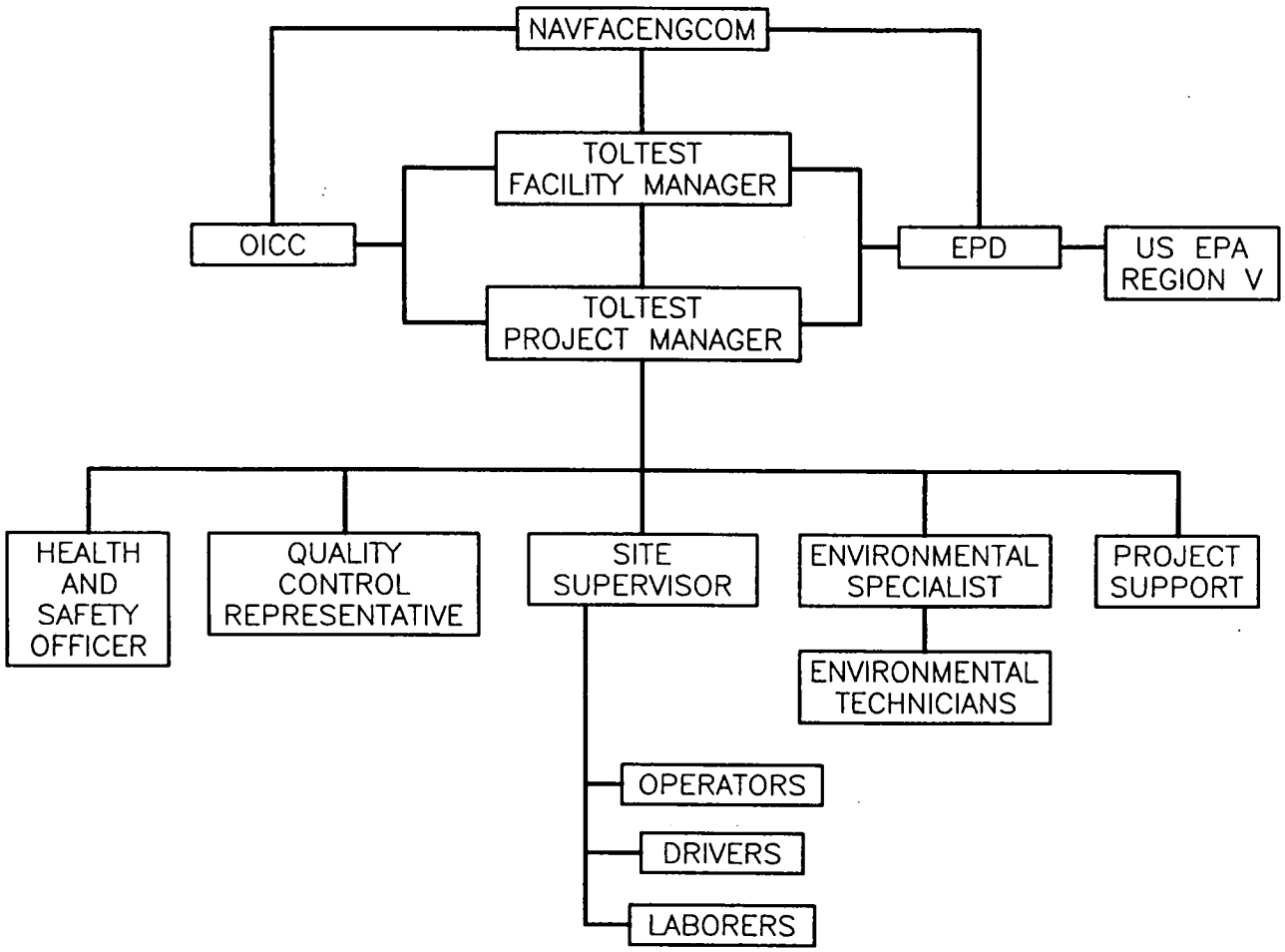

FIGURE 3

PROJECT ORGANIZATIONAL CHART

BATTERY SITE CLEAN UP
NAVAL SURFACE WARFARE CENTER, MINE FILL A
CRANE, INDIANA

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REVISED	APPROVED
JOB NO. 37826.01	TOLTEST, INC.
DRAWING NUMBER 378263	

FIGURE 4

SCHEDULE TIME LINE WILL BE SUBMITTED AFTER APPROVAL OF THE WORK PLAN

APPENDIX A

SITE SAFETY AND HEALTH PLAN

FINAL

SITE SAFETY AND HEALTH PLAN

SWMU SITE 12/14
MINE FILL A BATTERY SITE CLEANUP
NAVAL SURFACE WARFARE CENTER CRANE, INDIANA

ENVIRONMENTAL JOB ORDER CONTRACT
CONTRACT NO. N68950-96-D-0052
DELIVERY ORDER # FC04
TOLTEST PROJECT NO. 37826.01

Submitted to:

OFFICER IN CHARGE OF NAVFAC CONTRACTS
NAVAL SURFACE WARFARE CENTER
BUILDING 2516
300 HIGHWAY 361
CRANE, INDIANA 47522-5082

Submitted by:

TOLTEST, INC.
1915 NORTH 12TH STREET
P.O. BOX 2186
TOLEDO, OHIO 43603
(419) 241-7175

AUGUST 2000

Reviewed By:

Richard L. Barcum, CSP, CHMM Manager, Corporate Health and Safety	Date
Lance Parsons Sr. Project Manager	Date

TABLE OF CONTENTS

Page No.

SSHP Acknowledgement ..... A-ii

1.0 INTRODUCTION A-1

2.0 APPLICABILITY A-2

3.0 SITE SAFETY AND HEALTH A-3
- 3.1 Key Personnel A-3
- 3.2 Personal Protective Equipment A-4
- 3.3 Site Control Measures A-5
- 3.4 Site Standard Operating Safety Procedures A-5
- 3.5 Site-Specific Respiratory Protection A-7
- 3.6 Material Safety Data Sheets (MSDS) A-7

4.0 ACCIDENT PREVENTION A-8
- 4.1 Daily Safety Meetings A-8
- 4.2 Daily Safety Inspections A-8
- 4.3 Accident Reporting A-8
- 4.4 Excavation Safety A-8

5.0 EMERGENCY RESPONSE A-10
- 5.1 Work Zones and Evacuation Procedures A-10
- 5.2 Decontamination A-10
- 5.3 Emergency Medical Treatment and First Aid A-11
  - 5.3.1 Cold Stress A-11
  - 5.3.2 Heat Stress A-13
- 5.4 Emergency Alerting and Response Procedures A-15
- 5.5 Spill and Discharge Control A-18

ATTACHMENTS
- ATTACHMENT A Material Safety Data Sheets
- ATTACHMENT B Daily Safety Log
- ATTACHMENT C Equipment Inspection Form
- ATTACHMENT D Incident Forms
- ATTACHMENT E Directions and Maps to Area Hospitals

SSHP Acknowledgement

Project Name: SWMU 12/14, Battery Disposal Site, NSWC Crane, Indiana
Project Number: 37826.01

Declaration of Understanding

I have read and understand this Site Safety and Health Plan (SSHP) and agree to abide by the procedures and limitations specified within.

Name	Signature	Employer	SSN	Date
Peter J. Chevalier	[illegible]	TolTest	322-58-0716	2/14/01

1.0 INTRODUCTION

TolTest is responsible for the safety, health and emergency response provisions for the soil and debris removal, disposal and site restoration at SWMU 12/14 Mine Fill A Battery Disposal Site, Naval Surface Warfare Center (NSWC), Crane, Indiana. These provisions are provided through the development and implementation of TolTest's Corporate Health and Safety Plan, the Work Plan and this Site Safety and Health Plan (SSHP). All personnel on site, contractors and subcontractors included, will be informed of this plan and any potential health and safety hazards of the operation. All personnel scheduled to work on this site will complete the Declaration of Understand on page ii before commencing cleanup operations.

2.0 APPLICABILITY

This plan will be followed during all site activities starting with site mobilization through and including site demobilization. This plan incorporates the requirements of the following regulations and/or appropriate guidance:

- Federal Acquisition Regulation (FAR) clause 52.236-13, Accident Prevention,
- Occupational Safety and Health Administration (OSHA) Construction Industry Standards, 29 CFR 1926,
- OSHA General Industry Standards, 29 CFR 1910 (including but not limited to 29 CFR 1910.120, Hazardous Waste Site Activities),
- National Institute of Occupational Safety and Health (NIOSH)/OSHA/United States Coast Guard (USCG)/Environmental Protection Agency (EPA), Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities,
- 49 CFR Part 178
- OPNAVINST 5090.1B and the base Hazardous Waste Management Plan
- Other applicable Federal, State, and local safety and health requirements.

The implementation of the Work Plan includes furnishing all labor, transportation, supervision, material, and equipment and performing all operations in connection with the excavation, sampling and disposal of approximately 100 tons of soil and battery fragments along with site restoration. This site has been historically used as a dumping area for batteries and the potentially contaminated soil area has an unknown origin.

3.0 SITE SAFETY AND HEALTH

This section addresses the responsibilities for safety and health oversight, personnel protective equipment, site specific control measures, and operating procedures.

3.1 Key Personnel

The Site Safety and Health Officer (SSHO) implements and enforces the project safety program and procedures. The SSHO, or designee, will conduct the daily safety meetings and will interface as required with other site representatives. The SSHO performs duties such as confirming personnel are fit for duty, coordinating emergency medical care, conducting daily site safety inspections, and inspecting health and safety equipment. In addition, the SSHO is responsible for maintaining safety equipment, conducting and posting daily air monitoring results, conducting monitoring for thermal stress as site conditions warrant, providing site orientation safety training for all personnel actively involved in site work, and other site safety documentation. The SSHO takes the following actions when deemed appropriate.

- Orders immediate shutdown of site activities in the case of a medical emergency or unsafe practice.
- Ensures protective clothing and equipment are properly stored, used, and maintained.
- Ensures that the environmental and personnel monitoring operations are ongoing in accordance with technical specifications and required procedures.

The SSHO restricts visitors from areas of potential exposure to harmful substances. A safety log will be kept for all TolTest activities. This log will include daily safety meeting topics, training provided, air-monitoring information, visits from all outside personnel and any incidents related to health and safety.

The TolTest NSWC Facility Manager (FM) communicates directly with the designated Navy representative and will serve as the primary point of contact. The FM is responsible for determining the extent and level of input required for technical issues that arise during the tenure of the project. The FM ensures that all imminent danger situations, as defined by Section 13(a) of the Occupational Safety and Health Act (OSHAct), are mitigated immediately upon discovery. Serious violations of safety standards will be addressed immediately and mitigated as soon as possible. The FM is

responsible for reporting such imminent danger hazards and serious regulatory violation to the SSHO and Corporate Safety and Health Manager.

The Site Supervisor is responsible for all field activities and enforces safe work practices by all crewmembers. The Site Supervisor watches for ill effects in any of the crewmembers, especially those symptoms potentially caused by heat stress and chemical exposure. This individual oversees the safety of any visitors who enter the site.

The FM Site Supervisor and SSHO will investigate all accidents, with assistance from the SSHO.

The Corporate Safety and Health Manager is responsible for the following:

- Serves as the primary contact to resolve complex health and safety matters that may arise.
- Approves new or revised written health and safety-related procedures and safe work practice instructions.
- Assists in the investigation of significant incidents.
- Conducts periodic audits for compliance with the SSHP, OSHA, and TolTest Health and Safety regulations and policies.

Although the employer is responsible for providing a safe and healthful workplace, each employee is responsible for his/her own safety as well of the safety of co-workers. The employee shall use all equipment provided in a safe and responsible manner as directed by their supervision. Each employee is responsible for reporting any injuries, incidents, and safety infractions to their supervision and the SSHO so treatment can be obtained and/or corrective action taken.

3.2 Personal Protective Equipment

Personal protective equipment (PPE) is to be used by employees for each of the site tasks and operations being performed. The type of PPE will depend upon the level of potential exposure to hazards. Based on the Statement of Work (SOW) dated April 13, 1999, the chemicals of concern will be identified during the site pre-characterization. Samples will be analyzed for metals, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), pesticides, and explosives. During excavation and loading of contaminated soil, engineering controls,

such as wetting the soil will be utilized in order to minimize dust. Level D PPE is anticipated to be used for this site. If unexpected conditions arise or pre-characterization activities indicate that a higher level of protection is needed, PPE will be upgraded. Level D may include:

- Coveralls
- Gloves (optional)
- Boots/shoe, leather or chemical resistant, steel toe and shank
- Boots (outer), chemical resistant, disposable (optional)
- Hard hat
- Safety glasses or chemical splash goggles, impact resistant
- Hearing protection if noise level exceeds 85 dbs

3.3 Site Control Measures

Control procedures will be implemented to prevent unauthorized access to the work area. This will included safety cones and caution tape or barricades around the area. At the direction of the Navy, further site controls may be required to prevent trespassers from having access to the site.

The SSHO will ensure that all personnel entering the site have the necessary training and medical approval documentation. Personnel entering the site will be given a thorough briefing on the site hazards and safe work procedures prior to proceeding. All visitors will be expected to comply with applicable federal, state and local regulatory requirements as well as the requirements of this SSHP. Visitors will also be expected to provide their own protective equipment. In the event that a visitor does not adhere to the provisions of this SSHP, they will be requested to leave the work area. All non-conformance incidents will be recorded in the daily safety log. The SSHO will document a written record of all personnel entering and exiting the site.

3.4 Site Standard Operating Safety Procedures

The following safety rules shall be adhered to during all site activities:

- At least one copy of this plan will be available at the project site, in a location readily available to all personnel, including visitors.

- Personnel should practice contamination avoidance. All liquid, sludge, and soil samples will be collected in such a manner to minimize contact or exposure to the materials being sampled.

- No foods, beverages, or tobacco products shall be present or consumed in the Exclusion or Contamination Reduction Zones.

- No alcohol or drugs shall be present or consumed on site, or in any company vehicle. No personnel will be permitted to work while under the influence of alcohol or drugs while on site or operating a company vehicle.

- Emergency equipment will be located in the company vehicle in a readily accessible location. Emergency equipment will consist of fire extinguishers, first aid kit, mobile telephone, and emergency phone numbers.

- Contact shall be maintained between crew members at all times, and crew members must observe each other for signs of exposure to chemical, biological, or physical agents. Indications of adverse effects include, but are not limited to:

  - Changes in complexion and skin coloration;
  - Changes in coordination;
  - Changes in demeanor;
  - Excessive salivation and pupillary response; and
  - Changes in speech pattern.

- All personnel shall inform their partners or team members of non-visible effects of overexposure to chemical, biological, or physical agents. Symptoms of overexposure may include:

  - Headaches;
  - Dizziness;
  - Nausea;
  - Blurred vision;
  - Cramps; and
  - Irritation of the eyes, skin, or respiratory tract.

3.5 Site-Specific Respiratory Protection

During this project, soil impacted with various contaminants will be excavated. This may pose an inhalation hazards at the site. Depending upon the analytical results of the pre-characterization samples, the breathing zone of on-site workers may be monitored for specific chemical constituents. Engineering controls, such as wetting the work area, will be implemented in order to reduce fugitive dust and inhalation hazards. It is anticipated that respiratory protection will not be needed. If respiratory protection is deemed necessary, work will stop until the situation is assessed. The selection of respirators as well as any decisions regarding upgrading or downgrading of respiratory protection will be made by the SSHO, through consultation with the Corporate Safety and Health Manager.

3.6 Material Safety Data Sheets (MSDS)

The only known hazardous materials anticipated on site include metals, unleaded gasoline, diesel fuel, various oils, and lubricants. MSDS' for hazardous materials brought on site are included in Attachment A.

3.7 Field Training

Field personnel will be required to complete the following training prior to working on this site:

- Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste and Emergency Response Training per 29 Code of Federal Regulations (CFR) 1910.120, updated by annual 8-hour refresher training.
- OSHA 8-hour supervisor's training course in Hazardous Waste Site Safety per 29 CFR 1910.120(e) (field management personnel only).
- Site specific training.
- Respiratory training.
- Two on-site personnel will be required to have First Aid and CPR training.

Training will be verified as part of TolTest's review process.

4.0 ACCIDENT PREVENTION

TolTest will implement accident prevention procedures. This section includes a description of daily safety meetings, daily safety inspections, accident reporting and excavation safety.

4.1 Daily Safety Meetings

Daily safety meetings will be conducted by the SSHO, or designee. These meetings will take place at the beginning of each day or if there is a change in personnel. The scope of work will be discussed along with physical and chemical hazards, protective clothing and equipment, emergency procedures and directions to the hospital. The activity hazard analysis (AHA) associated with each major task will also be discussed (AHAs are presented in the Work Plan as Appendix D). The daily safety meeting will be documented on the Daily Safety Log (Attachment B). All attendees will sign the log.

4.2 Daily Safety Inspections

All machinery and equipment will be inspected and tested daily by the SSHO to ensure a safe operating condition. Inspections will be in accordance with the manufacturer's recommendations and will be documented. Records of inspections will be maintained at the site, made available upon request, and become part of the project file. The equipment inspection form can be found in Attachment C.

4.3 Accident Reporting

All reporting and record keeping requirements will be adhered to. TolTest's forms will be completed for all incidents including personal injury reports, safety incident reports, equipment damage reports, and vehicle accident reports. All reports will be submitted to the U.S. Navy's representative within 24 hours of any accident. Copies of these forms are included in Attachment D.

4.4 Excavation Safety

All excavating work will be conducted in strict conformance with, at a minimum, 29 CFR 1926.650 through 26 CFR 1926.653, including requirements for continuously sloping excavations to 1-1/2 to 1 (33°41') angle of repose. The excavated areas are anticipated

to be a depth of approximately two feet below grade and therefore the use of shoring and sheeting is not anticipated.

Notification will be made to the facility caretaker and Indiana Underground service 72 hours prior to excavation activities. Utility maps of the area surrounding the site will be examined. Caution will be used during excavation, as exact placements of buried services may not be known. Should undocumented utilities be encountered, the Contracting Officers Representative (COR) will be contacted immediately to facilitate relocations or possible interruptions. An unexploded ordnance clearance will be secured from the NSWC Crane Explosives Safety Officer before commencing excavation activities.

Clearances to adjacent overhead transmission and distribution electrical lines will be sufficient for the movement of vehicles and operation of construction equipment. TolTest will follow the requirements stated in OSHA 29 CFR 1926 General Construction Industry Standard and the National Electric Safety Code.

During periods when the work site is unoccupied (i.e., overnight, weekends and other similar off periods) safety cones and caution tape or barricades will be placed around the excavation site in such a manner to alert personnel to the danger and prevent them from entering the work area.

5.0 EMERGENCY RESPONSE

TolTest will implement an emergency response and contingency procedures, in accordance with OSHA standards 29 CFR 1910.120(L). This section addresses work zones and excavation procedures, decontamination, emergency medical treatment, emergency response procedures, spill and discharge control.

5.1 Work Zones and Evacuation Procedures

Daily safety meetings will identify the work zones for construction activities. The three general work zones established at the site are the exclusion zone (EZ), contamination reduction zone (CRZ), and support zone (SZ). The EZ is defined as the area where contamination is either known or likely to be present, or because of activity, will provide a potential to cause harm to personnel. Entry into the EZ requires the use of personnel protective equipment. Safety cones and caution tape or barricades will surround this zone.

The CRZ is the area where personnel conduct personal and equipment decontamination. It is essentially a buffer zone between contaminated areas and clean areas. The SZ is situated in clean areas where the chance to encounter hazardous materials or conditions is minimal. Safe distances are outside the EZ and places of refuge are outside the CRZ.

In the event of an emergency, which necessitates evacuation of the site, all personnel will be expected to leave the work zone, and mobilize at a safe distance, in an area using the designated evacuation routes. Personnel will remain at that area until the SSHO, Site Supervisor or designee provides further instructions.

5.2 Decontamination

All site personnel should minimize contact with contaminants to minimize the need for extensive decontamination. The SSHO is responsible for monitoring decontamination procedures and determining their effectiveness. Non-disposable PPE, sampling equipment, and heavy equipment will be decontaminated as describe in Section 3.8 of the Work Plan. Decontamination fluids will be containerized and sampled for disposal characterization.

5.3 Emergency Medical Treatment and First Aid

All TolTest employees participate in TolTest's medical screening and surveillance programs. If an injury/illness or exposure occurs, employees must seek medical attention immediately. TolTest field personnel are trained in first aid and CPR and can administer immediate assistance.

5.3.1 Cold Stress

Cold and/or wet environmental conditions can place workers at risk of cold-related illness. Hypothermia can occur whenever temperatures are below 45° F. The principal cause of hypothermia in these conditions usually involves the loss of insulating properties of clothing due to moisture; heat loss due to increased air movement, and evaporation of moisture on the skin.

Frostbite, the other illness associated with cold exposure, is the freezing of body tissue which ranges from superficial freezing of surface skin layers to deep freezing of underlying tissue. Frostbite will only occur when the ambient temperatures are below 32° F. The risk of frostbite increases as the temperature drops and the wind speed increases.

Site workers should be protected from exposure to cold so that the core body temperature does not fall below 98.6° F. Lower body temperatures will very likely result in reduced mental alertness, reduction in rational decision making, or loss of consciousness with the threat of fatal consequences. To prevent such occurrence, the following measures may be implemented:

- Site workers should wear warm clothing, such as gloves, heavy socks, etc., when the air temperature is below 45° F. Protective clothing, such as Tyvek or other disposable overall, may be used to shield employees from the wind.

- When the air temperature is below 32° F, clothing for warmth may include:

  - Insulated suits, such as whole body thermal underwear
  - Wool socks or polypropylene socks to keep moisture off the feet
  - Insulated gloves
  - Insulated boots

- Insulated head cover such as hard hat, winter liner, or knit cap
- Insulated jacket, with wind and water resistant outer layer

- At air temperatures below 32° F, the following work practices must be implemented:

  - If the clothing of a site worker might become wet in any way on the job site, the outer layer of clothing must be water permeable
  - If a site worker's underclothing becomes wet in any way, the worker must change into dry clothing immediately. If the clothing becomes wet from sweating (and the employee is uncomfortable), the employee may finish the task at hand prior to changing into dry clothing.
  - Site workers will be provided with a warm (65° F or above) break area.
  - The intake of coffee and tea should be limited, due to their circulatory and diuretic effects.
  - The buddy system shall be practiced at all times on site. Any site worker observed with severe shivering shall leave work area immediately.
  - Site workers should dress in layers, with thinner lighter clothing worn next to the body.

Frostbite. To administer First Aid for frostbite, take the exposed employee indoors and rewarm the areas quickly in warm water that is between 102° and 105° F, for about 20 minutes or until the frozen tissue regains the original color. Provide warms drinks, but no coffee, tea, or alcohol. Keep the affected (frozen) tissue in warm water or covered with warm clothing for 30 minutes, even though the tissue may be very painful as it thaws. If present, do not allow blisters to be broken. Use sterile, soft dry material to cover the injured area, keep the patient warm and get medical attention.

- Do not rub the frostbitten part (this may cause gangrene).
- Do not use heat lamps or hot water bottles to warm the frostbitten areas.
- Do not place the exposed part near a hot stove.

Hypothermia. Hypothermia is defined as a lowering of the core body temperature. General hypothermia, the more life-threatening cold injury, affects the entire body system. Once the body temperature is lowered to 95° F, thermal

control is lost, and the body is no longer in thermal balance. A coma may occur when the core temperature reaches to below 95° F. Death can occur within two hours of the first signs and symptoms. The general symptoms of the hypothermia are usually exhibited in five stages:

- Shivering;
- Apathy, listlessness, sleepiness, and (sometimes) rapid cooling of the body;
- Unconsciousness, glassy stare, slow pulse, and respiratory rate;
- Freezing of the extremities; and
- Death.

Extremely low temperatures are not necessary to induce hypothermia--it can occur in temperatures as high as 65° F, depending on the wind chill factor. Wind increases the body's heat loss by dispersing layers of warm air trapped between layers of clothing and skin. This heat loss increases as the wind speed increases.

5.3.2 Heat Stress

Physical hazards may involve heat-related symptoms such as heat stress, heat cramps, heat exhaustion, or heat stroke.

Heat stress is the aggregate of environmental and physical work factors that make up the total heat load imposed on the body. The environmental factors of heat stress include air temperature, humidity, radiant heat exchange, and wind/water vapor pressure (related to humidity). Physical work contributes to the total heat stress by producing metabolic heat in the body, proportional to the intensity of the work. Heavy physical labor can greatly increase the likelihood of heat fatigue, heat exhaustion, and heat stroke, the latter being a life threatening condition. Heat stress monitoring and observation of personnel shall commence when the ambient temperature is 80° F or above (65° F, if chemical protective clothing is worn).

All employees will be informed of the possibility and symptoms of heat stress. If an employee experiences extreme fatigue, cramps, dizziness, headache, nausea, profuse sweating, or pale, clammy skin, the employee and the SSHO will take control measures.

If the symptoms do not subside after a reasonable rest period, the SSHO shall seek medical assistance.

To prevent heat stress, the following control measures will be implemented.

- Site workers will be encouraged to drink plenty of water throughout the day.
- On-site drinking water will be kept cool to encourage personnel to drink frequently.
- A work regimen that will provide adequate rest periods for cooling down will be established, as required.
- All personnel will be advised of the dangers and symptoms of heat stroke, heat exhaustion, and heat cramps.
- Employees should be instructed to observe and monitor themselves and coworkers for signs of heat stress and to take additional breaks as necessary.
- All breaks should take place in cool, well-ventilated, and shaded rest areas.

Heat Cramps

Heat cramps are caused by heavy sweating and inadequate electrolyte replacement. Symptoms include muscle spasms and pain in the hands, feet or abdomen.

Heat Exhaustion

Heat exhaustion occurs from increased stress on various body organs. Signs and symptoms include:

- Pale, cool, moist skin
- Heavy sweating
- Dizziness, nausea
- Fainting

Heat Stroke

Heat stroke is the most serious form of heat stress and should always be treated as a medical emergency. The body's temperature regulation system fails and the body temperature rapidly rises to critical levels. Immediate action must be taken to cool the body before serious injury or death occurs. Signs and symptoms of heat stroke include:

- Red, hot, usually dry skin
- Lack of or reduced respiration
- Nausea
- Dizziness and confusion
- Strong, rapid pulse and confusion
- Coma

The differential diagnosis for heat stroke is the lack of sweating as the body's defense mechanisms for shedding excess heat fail.

5.3.3 Flora and Fauna Hazards

Insects, animals, and plants which can cause an adverse health effect on workers are a concern at this site. Stings, bites, and allergic reactions to certain plants can cause localized swelling and itching which can be treated with basic first aid practices. The SSHO should be notified if an individual has a known allergy to any specific insect or allergen. Insect repellant may be used to minimize the potential for bites and stings. In the event of an allergic reaction, proper medical attention will be sought immediately.

Bites from ticks are common to this area and precautions will be taken to identify and train workers on the signs and symptoms of Lymes disease. Warning signs for this disease are a rash which can lead into fever, headache, weakness, and joint and muscle pain similar to the flu. Workers will be trained on the proper method to remove a tick and treatment of the bite area.

Encounters with wild animals (e.g. mammals) are not expected but the possibility for contact with snakes is high given the nature of the site (tall grass, woods, large rocks). Workers will be warned of this potential and appropriate precautions will be taken during sampling, clearing, and excavation activities.

5.4 Emergency Alerting and Response Procedures

All hazardous substance releases or spills involving Government waste or Government property, other than the original release, will be immediately reported to the US Navy. Spill cleanup and remediation and damage to the environment resulting from TolTest actions will be the responsibility of TolTest.

Below are a list of emergency numbers, emergency service organizations and directions to the nearest hospitals. A telephone is located inside the TolTest support truck.

Emergency Telephone Numbers

Jim Hunsicker or Tom Brent	NSWC Environmental Protection Services	(812) 854-6160
Law Enforcement	NSWC Security (Base)	3300 emergency
Fire Department	NSWC Fire Department (Base)	3300 emergency (812) 854-1235
Ambulance Service	NSWC Ambulance (Base)	3300 emergency (812) 854-1100
Dale Groh	NSWC Safety Director	(812) 854-3601
Rich Barcum, CSP	TolTest Corp. Safety Manager	(419) 241-7175
		(419) 460-7655 - cell
		(800) 680-1071 - pager
Poison Control Center	Poison Control Center	(800) 942-5969
National Response Center	National Response Center	(800) 424-8802
Richard Karl	Regional Chemical Accident Response National Response Team	(312) 353-9295
Medical Facility (Non-Life Threatening Injuries)	Prompt Care	(812) 332-3443
Medical Facility (Life-Threatening Injuries ONLY)	NSWC Base Medical Facility Head Nurse (Mary Muessig)	(812) 854-1220 (812) 854-4319
Hospital	Bedford Medical Center 2900 West 16th Street Bedford, IN 47432	(812) 275-1200
Hospital	Bloomington Hospital 601 West Second Street Bloomington, IN	(812) 336-9515
Utility Locator Service	Public Works Dept.	(812) 854-1834

Directions to NSWC Medical Facility On-site:

The NSWC Fire Department coordinates the on-site ambulance service. The Medical Facility is located in Building 12, off of H-2 just north of H-5. This location will be used

for life threatening emergencies only.

Directions to Bedford Medical Center:

From Bloomington Gate, head east on Highway 58 to the city of Bedford, then turn left onto 16th Street. Distance to hospital is approximately 20 miles.

Directions to Bloomington Hospital:
Exit NSWC Crane on H5-45 through the Bloomington Gate, then follow Highway 45 North to Bloomington. At the intersection of Highway 45 and Highway 37, continue straight ahead over the bypass (Bloomfield Road), and follow Bloomfield Road north until it becomes 2nd street. Continue on 2nd street and the hospital will be on right hand side of the road.

Directions and area maps to the hospitals are located in Attachment E.

5.5 Spill and Discharge Control

This section provides contingency measures for potential spills and discharges from the handling and transportation of any contaminated soil, contamination fluids, and/or oil/fuel. If a spill or discharge occurs, the following actions, at a minimum, will be taken:

1. Notify the U.S. Navy representative immediately.

2. Take immediate measures to control and contain the spill within the site boundaries. This will include, at a minimum, the following actions:

   - Keep unnecessary people away, isolate hazardous areas, and deny entry.
   - Do not allow anyone to touch spilled material.
   - Stay upwind; keep out of low areas where fluids and/or vapors may accumulate.
   - Keep combustibles away from the spilled material
   - Use water spray or foam to reduce vapor or dust generation, as needed
   - Take samples for analysis to determine that clean up is adequate
   - Take other corrective measures, as needed

A written report will be submitted to the U.S. Navy within seven days of a verbal report. The SSHO will conduct spill prevention briefings daily during safety meetings for all personnel who are involved with handling, receipt, storage, and/or cleanup of oil/fuel.

Storage - All tanks, containers, and pumping equipment used for the storage or handling of flammable and combustible liquids will be labeled or placarded in accordance with the U.S. Department of Transportation. Oils or fuels temporarily stored will be kept in tightly sealed containers, with the exception of proper venting, in fire-resistant areas and at safe distances from ignition sources. All transfer vessels will be emptied at the end of the workday.

Pumping Flammable and Combustible Liquids - Flammable liquid pumping systems will be electrically bonded and grounded, and will be drawn from, or transferred into vessels, containers, or tanks through a closed piping system, from safety cans, by means of a device drawing through the top, or from a container, or portable tanks, by gravity or pump, through an approved self closing valve. Transferring by means of air pressure on the container or portable tank is prohibited.

Equipment Inspection - Equipment inspection is part of the daily routine during field activities. The Site Supervisor is to ensure that no oil/fuel spill has accumulated in any area by conducting daily visual inspection of the equipment. Equipment and safety issues will be documented in the daily report.

ATTACHMENT A

Material Safety Data Sheets

69431

SECTION 1 CHEMICAL PRODUCTS & COMPANY IDENTIFICATION

OCCUPATIONAL HEALTH SERVICES, INC.
11 WEST 42ND STREET, 12TH FLOOR
NEW YORK, NEW YORK 10036
1-800-445-MSDS (1-800-445-6737) OR
1-212-789-3535

FOR EMERGENCY SOURCE INFORMATION
CONTACT: 1-615-366-2000 USA

CAS NUMBER: 72623-87-1

SUBSTANCE: LUBRICATING OILS

TRADE NAMES/SYNONYMS:
LUBRICATING OILS (PETROLEUM), C20-50, HYDROTREATED NEUTRAL OIL BASED; SEVERELY HYDROTREATED PARAFFINIC OIL (C23-35); LUBE OIL; OHS69431

CHEMICAL FAMILY:
Petroleum hydrocarbon

CREATION DATE: 03/29/93 REVISION DATE: 09/10/93

SECTION 2 COMPOSITION/INFORMATION ON INGREDIENTS

COMPONENT : LUBRICATING OILS
CAS NUMBER: 72623-87-1
PERCENTAGE: 100.0

OTHER CONTAMINANTS: NONE.

SECTION 3 HAZARDS IDENTIFICATION

CERCLA RATINGS (SCALE 0-3): HEALTH=1 FIRE=1 REACTIVITY=0 PERSISTENCE=0
NFPA RATINGS (SCALE 0-4): HEALTH=1 FIRE=1 REACTIVITY=0

EMERGENCY OVERVIEW:
Light colored, viscous liquid with a hydrocarbon odor.
May be irritating to the respiratory tract, skin and eyes. May damage the lungs.
Avoid breathing vapor or mist. Avoid contact with eyes, skin and clothing. Keep container tightly closed. Wash thoroughly after handling. Use only with adequate ventilation.

POTENTIAL HEALTH EFFECTS:
INHALATION:
SHORT TERM EFFECTS: May cause irritation.
LONG TERM EFFECTS: May cause lung damage.
SKIN CONTACT:
SHORT TERM EFFECTS: May cause irritation.
LONG TERM EFFECTS: In addition to effects from short term exposure, acne may

occur.
E CONTACT:
ORT TERM EFFECTS: May cause irritation.
NG TERM EFFECTS: No information is available.
INGESTION:
SHORT TERM EFFECTS: No information available on significant adverse effects.
LONG TERM EFFECTS: No information is available.

CARCINOGEN STATUS:
OSHA: N
NTP: N
IARC: N

SECTION 4 FIRST AID MEASURES

INHALATION:
FIRST AID- Remove from exposure area to fresh air immediately. If breathing has stopped, perform artificial respiration. Keep person warm and at rest. Treat symptomatically and supportively. Get medical attention immediately.

SKIN CONTACT:
FIRST AID- Remove contaminated clothing and shoes immediately. Wash affected area with soap or mild detergent and large amounts of water until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

EYE CONTACT:
FIRST AID- Wash eyes immediately with large amounts of water or normal saline, occasionally lifting upper and lower lids, until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

INGESTION:
FIRST AID- Treat symptomatically and supportively. Get medical attention immediately. If vomiting occurs, keep head lower than hips to prevent aspiration.

NOTE TO PHYSICIAN
ANTIDOTE:
No specific antidote. Treat symptomatically and supportively.

SECTION 5 FIRE FIGHTING MEASURES

FIRE AND EXPLOSION HAZARD:
Slight fire hazard when exposed to heat or flame.

EXTINGUISHING MEDIA:
Dry chemical, carbon dioxide, water spray or regular foam
(1990 Emergency Response Guidebook, DOT P 5800.5).

larger fires, use water spray, fog or regular foam
Emergency Response Guidebook, DOT P 5800.5).

FIREFIGHTING:
Move container from fire area if you can do it without risk. Do not scatter spilled material with high-pressure water streams. Dike fire-control water for later disposal (1990 Emergency Response Guidebook, DOT P 5800.5, Guide Page 31).

Use agents suitable for type of surrounding fire. Avoid breathing hazardous vapors, keep upwind.

FLASH POINT: 392-406 F (200-208 C) (COC)
AUTOIGNITION: >671 F (>355 C)
FLAMMABILITY CLASS(OSHA): IIIB

HAZARDOUS COMBUSTION PRODUCTS:
Thermal decomposition products may include toxic oxides of carbon.

SECTION 6 ACCIDENTAL RELEASE MEASURES

OCCUPATIONAL SPILL:
Stop leak if you can do it without risk. For small spills, take up with sand or other absorbent material and place into clean, dry containers for later disposal. Keep unnecessary people away. Isolate hazard area and deny entry.

SECTION 7 HANDLING AND STORAGE

Observe all federal, state and local regulations when storing this substance.

Store in a cool, well-ventilated area.

Store away from incompatible substances.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

EXPOSURE LIMITS:
MINERAL OIL MIST:
5 mg/m3 OSHA TWA
5 mg/m3 ACGIH TWA; 10 mg/m3 ACGIH STEL
(Notice of Intended Changes 1993-94)
5 mg/m3 NIOSH recommended TWA;
10 mg/m3 NIOSH recommended STEL

Measurement method: Particulate filter;
1,1,2-trichloro-1,2,2-trifluoroethane; infrared spectrometry;
(NIOSH Vol. III # 5026).

VENTILATION:
Provide local exhaust ventilation system to meet published exposure limits.

EYE PROTECTION:
Employee must wear splash-proof or dust-resistant safety goggles to prevent

'e contact with this substance.

(ɟency eye wash: Where there is any possibility that an employee's eyes may e ɔxposed to this substance, the employer should provide an eye wash ountain within the immediate work area for emergency use.

LOTHING:
ear oil impervious clothing. Avoid prolonged or repeated contact with ubstance. Avoid wearing oil soaked clothing.

LOVES:
mployee must wear appropriate protective gloves to prevent contact with this ubstance.

ESPIRATOR:
he following respirators and maximum use concentrations are recommendations by the U.S. Department of Health and Human Services, NIOSH Pocket Guide to Chemical Hazards; NIOSH criteria documents or by the U.S. Department of Labor, 29 CFR 1910 Subpart Z.
he specific respirator selected must be based on contamination levels found in the work place, must not exceed the working limits of the respirator and be jointly approved by the National Institute for Occupational Safety and Health and the Mine Safety and Health Administration (NIOSH-MSHA).

INERAL OIL MIST:

50 mg/m3- Any supplied-air respirator.
Any self-contained breathing apparatus.
Any air-purifying respirator with a high-efficiency particulate filter.

125 mg/m3- Any supplied-air respirator operated in a continuous-flow mode.
Any powered, air-purifying respirator with a high-efficiency particulate filter.

250 mg/m3- Any air-purifying, full-facepiece respirator with a high-efficiency particulate filter.
Any powered, air-purifying respirator with a tight-fitting facepiece and a high-efficiency particulate filter.
Any supplied-air respirator that has a tight-fitting facepiece and is operated in a continuous-flow mode.
Any self-contained breathing apparatus with a full facepiece.
Any supplied-air respirator with a full facepiece.

500 mg/m3- Any supplied-air respirator operated in a pressure-demand or other positive-pressure mode.

Escape- Any air-purifying, full-facepiece respirator with a high-efficiency particulate filter.
Any appropriate escape-type, self-contained breathing apparatus.

OR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

(/ self-contained breathing apparatus that has a full facepiece and is

operated in a pressure-demand or other positive-pressure mode.

supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

DESCRIPTION: Light colored, viscous liquid with a hydrocarbon odor.
BOILING POINT: 714-889 F (379-476 C) (approximate)
VAPOR PRESSURE: <0.008 mmHg @ 25 C
SPECIFIC GRAVITY: 0.876 @ 15 C
WATER SOLUBILITY: negligible
VOLATILITY: 0%
VISCOSITY: 32 cSt @ 40 C
SOLVENT SOLUBILITY: Soluble in ether.

POUR POINT: 0 F (-18 C)

SECTION 10 STABILITY AND REACTIVITY

REACTIVITY:
Stable under normal temperatures and pressures.

TIONS TO AVOID:
May burn but does not ignite readily. Avoid contact with strong oxidizers, excessive heat, sparks, or open flame.

INCOMPATIBILITIES:
LUBRICATING OILS:
OXIDIZERS (STRONG): Fire and explosion hazard.

HAZARDOUS DECOMPOSITION:
Thermal decomposition products may include toxic oxides of carbon.

POLYMERIZATION:
Hazardous polymerization has not been reported to occur under normal temperatures and pressures.

SECTION 11 TOXICOLOGY INFORMATION

LUBRICATING OILS:
TOXICITY DATA: >5000 mg/kg oral-rat LD50 (Petro-Canada, Inc. MSDS)*.
CARCINOGEN STATUS: None.
ACUTE TOXICITY LEVEL: Slightly toxic by ingestion.
TARGET EFFECTS: Poisoning may affect the lungs.

* Estimated

H EFFECTS

'HALATION:
BRICATING OILS:
UTE EXPOSURE- May cause irritation of the respiratory tract.
RONIC EXPOSURE- Sprays and mists may cause chemical pneumonitis.

KIN CONTACT:
UBRICATING OILS:
ACUTE EXPOSURE- May cause irritation.
CHRONIC EXPOSURE- Repeated or prolonged contact may cause acneform lesions and dermatitis. Skin contact has rarely resulted in carcinoma.

YE CONTACT:
UBRICATING OILS:
ACUTE EXPOSURE- May cause irritation and smarting of the eyes.
CHRONIC EXPOSURE- No data available.

NGESTION:
UBRICATING OILS:
ACUTE EXPOSURE- May cause a laxative effects. Aspiration with subsequent pulmonary infiltration is possible.
CHRONIC EXPOSURE- No data available.

SECTION 12 ECOLOGICAL INFORMATION

NVIRONMENTAL IMPACT RATING (0-4): no data available

E AQUATIC TOXICITY: no data available

EGRADABILITY: no data available

OG BIOCONCENTRATION FACTOR (BCF): no data available

OG OCTANOL/WATER PARTITION COEFFICIENT: no data available

SECTION 13 DISPOSAL INFORMATION

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bserve all federal, state and local regulations when disposing of this substance.

SECTION 14 TRANSPORTATION INFORMATION

No classification currently assigned

SECTION 15 REGULATORY INFORMATION

SCA STATUS: Y

ERCLA SECTION 103 (40CFR302.4): N
SECTION 302 (40CFR355.30): N
SECTION 304 (40CFR355.40): N

SARA SECTION 313 (40CFR372.65): N
HAZ PROCESS SAFETY (29CFR1910.119): N
CALIFORNIA PROPOSITION 65: N

SARA HAZARD CATEGORIES, SARA SECTIONS 311/312 (40 CFR 370.21)

ACUTE HAZARD:	N
CHRONIC HAZARD:	N
FIRE HAZARD:	N
REACTIVITY HAZARD:	N
SUDDEN RELEASE HAZARD:	N

SECTION 16 OTHER

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340

SECTION 1 CHEMICAL PRODUCTS & COMPANY IDENTIFICATION

)CCUPATIONAL HEALTH SERVICES, INC.
.1 WEST 42ND STREET, 12TH FLOOR
IEW YORK, NEW YORK 10036
.-800-445-MSDS (1-800-445-6737) OR
.-212-789-3535

FOR EMERGENCY SOURCE INFORMATION
CONTACT: 1-615-366-2000 USA

CAS NUMBER: 8006-61-9
RTECS NUMBER: LX3373000

SUBSTANCE: GASOLINE, AUTOMOTIVE, UNLEADED

TRADE NAMES/SYNONYMS:
UNLEADED GASOLINE; PREMIUM UNLEADED GASOLINE; PETROL; MOTOR SPIRITS; BENZIN; GASOLINE; "A" GRADE GASOLINE (NCRA); "N" GRADE GASOLINE (NCRA); 420003415; 00000024; UN 1203; STCC 4908178; OHS10340

CHEMICAL FAMILY:
Petroleum hydrocarbon

CREATION DATE: 04/23/85 REVISION DATE: 01/15/94

TION 2 COMPOSITION/INFORMATION ON INGREDIENTS

COMPONENT : GASOLINE, AUTOMOTIVE, UNLEADED
CAS NUMBER: 8006-61-9
PERCENTAGE: 100.0
MAY CONTAIN:
BENZENE >0.1%
CAS NUMBER: 71-43-2

OTHER CONTAMINANTS: NONE

SECTION 3 HAZARDS IDENTIFICATION

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=3 REACTIVITY=0 PERSISTENCE=1
NFPA RATINGS (SCALE 0-4): HEALTH=1 FIRE=3 REACTIVITY=0

EMERGENCY OVERVIEW:
Clear colorless to amber, aromatic, volatile liquid
Cancer hazard (contains material which can cause cancer in humans). Risk of cancer depends on duration and level of exposure. Causes respiratory tract, skin and eye irritation. May cause blood disorders. May cause convulsions. May damage nerves. May affect the central nervous system. May cause adverse reproductive effects. May cause brain damage. May cause hearing loss. May affect the heart. May affect the kidneys. May affect the liver. May damage lungs. May cause visual disturbances. Flammable liquid and vapor. May cause flash fire.

Do not breathe vapor or mist. Do not get in eyes, on skin, or on clothing. eep away from all ignition sources. Keep container tightly closed. Wash oroughly after handling. Use only with adequate ventilation. Handle with ution.

POTENTIAL HEALTH EFFECTS:
INHALATION:
SHORT TERM EFFECTS: May cause irritation. Additional effects may include paleness, flushing, ringing in the ears, lack of appetite, nausea, vomiting, difficulty speaking, difficulty swallowing, chest pain, difficulty breathing, irregular heartbeat, headache, weakness, drowsiness, drunkeness, feeling of well-being, confusion, disorientation, nervousness, restlessness, sleeplessness, numbness, twitching, visual disturbances, suffocation, lung damage, blood disorders, nerve effects, paralysis, convulsions, shock, unconsciousness and coma.
LONG TERM EFFECTS: In addition to effects from short term exposure, weight loss, low blood pressure, loss of memory, hearing loss, bruising, kidney damage, nerve damage and brain damage may occur. May also cause reproductive effects and cancer.
SKIN CONTACT:
SHORT TERM EFFECTS: May cause irritation. Additional effects may include blisters, blood in the urine, low blood pressure, lung damage and kidney damage.
LONG TERM EFFECTS: In addition to effects from short term exposure, burns, tingling sensation and nerve effects may occur.
EYE CONTACT:
HORT TERM EFFECTS: May cause irritation. Additional effects may include astic winking.
NG TERM EFFECTS: In addition to effects from short term exposure, cataracts may occur.
INGESTION:
SHORT TERM EFFECTS: May cause gastrointestinal irritation. Additional effects may include coughing, paleness, flushing, fever, nausea, vomiting, diarrhea, chest pain, difficulty breathing, irregular heartbeat, headache, weakness, drunkeness, feeling of well-being, confusion, disorientation, nervousness, restlessness, excitation or drowsiness, twitching, visual disturbances, bluish skin color, suffocation, lung damage, liver damage, paralysis, convulsions, unconsciousness, coma and heart failure.
LONG TERM EFFECTS: In addition to effects from short term exposure, anemia and impotence may occur. May also cause reproductive effects and cancer.

CARCINOGEN STATUS:
OSHA: Y
NTP: Y
IARC: Y

SECTION 4 FIRST AID MEASURES

INHALATION:
FIRST AID- Remove from exposure area to fresh air immediately. If breathing s stopped, give artificial respiration. Maintain airway and blood essure and administer oxygen if available. Keep affected person warm and

at rest. Treat symptomatically and supportively. Administration of oxygen should be performed by qualified personnel. Get medical attention ediately.

SKIN CONTACT:
FIRST AID- Remove contaminated clothing and shoes immediately. Wash affected area with soap or mild detergent and large amounts of water until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

EYE CONTACT:
FIRST AID- Wash eyes immediately with large amounts of water or normal saline, occasionally lifting upper and lower lids, until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

INGESTION:
FIRST AID- Only hydrocarbons that are solvents for a toxic agent or are themselves toxic need to be evacuated. Extreme care must be used to prevent aspiration. Gastric lavage with a cuffed endotracheal tube in place to prevent further aspiration should be done within 15 minutes. In the absence of depression or convulsions or impaired gag reflex, emesis can also be induced using syrup of ipecac without increasing the hazard of aspiration (Dreisbach, Handbook of Poisoning, 12th Ed.). Treat symptomatically and supportively. Gastric lavage should be performed by qualified medical personnel. Get medical attention immediately.

E TO PHYSICIAN
OTE:
No ecific antidote. Treat symptomatically and supportively.

SECTION 5 FIRE FIGHTING MEASURES

FIRE AND EXPLOSION HAZARD:
Dangerous fire hazard when exposed to heat or flame.

Vapors are heavier than air and may travel a considerable distance to a source of ignition and flash back.

Vapor-air mixtures are explosive.

EXTINGUISHING MEDIA:
Dry chemical, carbon dioxide, water spray or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

For larger fires, use water spray, fog or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

FIREFIGHTING:
Move container from fire area if you can do it without risk. Apply cooling water to sides of containers that are exposed to flames until well after fire out. Stay away from ends of tanks. For massive fire in cargo area, use ned hose holder or monitor nozzles; if this is impossible, withdraw from and let fire burn. Withdraw immediately in case of rising sound from

nting safety device or any discoloration of tank due to fire. Isolate for mile in all directions if tank, rail car or tank truck is involved in fire) Emergency Response Guidebook, DOT P 5800.5, Guide Page 27).

xtinguish only if flow can be stopped; use water in flooding amounts as fog, olid streams may spread fire. Cool containers with flooding amounts of water, pply from as far a distance as possible. Avoid breathing vapors, keep pwind. Evacuate to a radius of 1500 feet for uncontrollable fires. Consider vacuation of downwind area if material is leaking.

ater may be ineffective (NFPA 325M, Fire Hazard Properties of Flammable iquids, Gases, and Volatile Solids, 1991)

LASH POINT: -45 F (-43 C) (CC)
OWER FLAMMABLE LIMIT: 1.2%
PPER FLAMMABLE LIMIT: 7.6%
UTOIGNITION: 536-853 F (280-456 C)
LAMMABILITY CLASS(OSHA): IB

AZARDOUS COMBUSTION PRODUCTS:
hermal decomposition products may include toxic oxides of carbon.

SECTION 6 ACCIDENTAL RELEASE MEASURES

CCUPATIONAL SPILL:
t off ignition sources. Stop leak if you can do it without risk. Use water to reduce vapors. For small spills, take up with sand or other absorbent a rial and place into containers for later disposal. For larger spills, dike ar ahead of spill for later disposal. No smoking, flames or flares in hazard rea. Keep unnecessary people away; isolate hazard area and restrict entry.

eportable Quantity (RQ):
he Superfund Amendments and Reauthorization Act (SARA) Section 304 requires hat a release equal to or greater than the reportable quantity established or that substance be immediately reported to the local emergency planning ommittee and the state emergency response commission (40 CFR 355.40). If the elease of this substance is reportable under CERCLA Section 103, the National esponse Center must be notified immediately at (800) 424-8802 or 202) 426-2675 in the metropolitan Washington, D.C. area (40 CFR 302.6).

ATER SPILL:
he California Safe Drinking Water and Toxic Enforcement Act of 1986 Proposition 65) prohibits contaminating any known source of drinking water ith substances known to cause cancer and/or reproductive toxicity.

SECTION 7 HANDLING AND STORAGE

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bserve all federal, state and local regulations when storing this substance.

ore in accordance with 29 CFR 1910.106.

away from incompatible substances.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

EXPOSURE LIMITS:
GASOLINE (BULK HANDLING):
300 ppm (900 mg/m3) OSHA TWA; 500 ppm (1,500 mg/m3) OSHA STEL
300 ppm (900 mg/m3) ACGIH TWA; 500 ppm (1,500 mg/m3) ACGIH STEL

BENZENE:
1 ppm OSHA TWA; 5 ppm OSHA 15 minute STEL; 0.5 ppm OSHA action level
10 ppm (30 mg/m3) ACGIH TWA;
ACGIH A2-Suspected Human Carcinogen
(Notice of Intended Changes 1990-91)
0.1 ppm (0.32 mg/m3) NIOSH recommended 8 hour TWA;
1 ppm (3.2 mg/m3) NIOSH recommended 15 minute ceiling

Measurement method: Charcoal tube; carbon disulfide; gas chromatography with flame ionization detection; (NIOSH Vol. III # 1500, Hydrocarbons).

10 pounds CERCLA Section 103 Reportable Quantity
Subject to SARA Section 313 Annual Toxic Chemical Release Reporting
Subject to California Proposition 65 cancer and/or reproductive toxicity warning and release requirements- (February 27, 1987)

OSHA revoked the final rule limits of January 19, 1989 in response to the 11th Circuit Court of Appeals decision (AFL-CIO v. OSHA) effective June 30, 1993. See 29 CFR 1910.1000 (58 FR 35338)

VENTILATION:
Provide local exhaust or general dilution ventilation to meet published exposure limits. Ventilation equipment should be explosion-proof if explosive concentrations of dust, vapor or fume are present.

EYE PROTECTION:
Employee must wear splash-proof or dust-resistant safety goggles to prevent eye contact with this substance.

Emergency eye wash: Where there is any possibility that an employee's eyes may be exposed to this substance, the employer should provide an eye wash fountain within the immediate work area for emergency use.

CLOTHING:
Employee must wear appropriate protective (impervious) clothing and equipment to prevent repeated or prolonged skin contact with this substance.

Any clothing wet with a flammable liquid should be immediately removed at the location where it is wetted to prevent burns from possible ignition.

GLOVES:
Employee must wear appropriate protective gloves to prevent contact with this substance.

RESPIRATOR:

following respirators are recommended based on information found in the physical data, toxicity and health effects sections. They are ranked in order from minimum to maximum respiratory protection.
The specific respirator selected must be based on contamination levels found in the work place, must be based on the specific operation, must not exceed the working limits of the respirator and must be jointly approved by the National Institute for Occupational Safety and Health and the Mine Safety and Health Administration (NIOSH-MSHA).

Any chemical cartridge respirator with organic vapor cartridge(s) and a full facepiece.

Any gas mask with organic vapor canister (chin-style or front- or back-mounted canister), with a full facepiece.

Any type 'C' supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure mode or with a full facepiece, helmet or hood operated in a continuous-flow mode.

Any self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.

FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode.

Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

DESCRIPTION: Clear colorless to amber, aromatic, volatile liquid
BOILING POINT: 100-400 F (38-204 C)
VAPOR DENSITY: 3.0-4.0
SPECIFIC GRAVITY: 0.7-0.8
WATER SOLUBILITY: insoluble
ODOR THRESHOLD: 0.25 ppm
SOLVENT SOLUBILITY: Absolute alcohol, ether, chloroform, benzene

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SECTION 10 STABILITY AND REACTIVITY

REACTIVITY:
Stable under normal temperatures and pressures.

CONDITIONS TO AVOID:
Avoid contact with heat, sparks, flames, or other sources of ignition. Vapors may be explosive. Avoid overheating of containers; containers may violently rupture in heat of fire. Avoid contamination of water sources.

COMPATIBILITIES:
GASOLINE, AUTOMOTIVE, UNLEADED:
OXIDIZERS (STRONG): Fire and explosion hazard.

HAZARDOUS DECOMPOSITION:
Thermal decomposition products may include toxic oxides of carbon.

POLYMERIZATION:
Hazardous polymerization has not been reported to occur under normal temperatures and pressures.

SECTION 11 TOXICOLOGY INFORMATION

GASOLINE, AUTOMOTIVE, UNLEADED:
(Data derived from unspecified and unleaded gasoline)
IRRITATION DATA: 500 mg/24 hours skin-rabbit mild; 500 ppm/1 hour eye-man moderate; 140 ppm/8 hours eye-human mild.
TOXICITY DATA: 900 ppm/1 hour inhalation-man TCLo; 300 gm/m3/5 minutes inhalation-rat LC50; 300 gm/m3/5 minutes inhalation-mouse LC50; 300 gm/m3/5 minutes inhalation-guinea pig LC50; 30000 ppm/5 minutes inhalation-mammal LCLo; 18,000 mg/kg oral-rat LD50; 13.6 gm/kg oral-rat LD50 (AETODY); 53 mg/kg parenteral-man TDLo; tumorigenic data (RTECS).
CARCINOGEN STATUS: Human Inadequate Evidence, Animal Limited Evidence (IARC Group-2B). In studies with mice and rats by inhalation, an increased incidence of hepatocellular adenomas and carcinomas was produced in female not male mice; an increased incidence of adenomas and carcinomas of the kidney was produced in male but not female rats.
LOCAL EFFECTS: Irritant- inhalation, skin, eye.
ACUTE TOXICITY LEVEL: Relatively non-toxic by inhalation and ingestion.
TARGET EFFECTS: Central nervous system depressant; simple asphyxiant.
ADDITIONAL DATA: The use of alcoholic beverages enhances the toxic effects. Stimulants such as epinephrine may induce ventricular fibrillation.

BENZENE:
IRRITATION DATA: 20 mg/24 hours skin-rabbit moderate; 15 mg/24 hours open skin-rabbit mild; 88 mg eye-rabbit moderate; 2 mg/24 hours eye-rabbit severe.
TOXICITY DATA: 2000 ppm/5 minutes inhalation-human LCLo; 2 pph/5 minutes inhalation-human LCLo; 65 mg/m3/5 years inhalation-human LCLo; 100 ppm inhalation-human TCLo; 150 ppm/1 year intermittent inhalation-man TCLo; 20,000 ppm/5 minutes inhalation-mammal LCLo; 10,000 ppm/7 hours inhalation-rat LC50; 300 ppm/6 hours/13 weeks intermittent inhalation-rat TCLo; 9980 ppm inhalation-mouse LC50; 300 ppm/6 hours/13 weeks intermittent inhalation-mouse TCLo; 103 ppm/6 hours/5 days intermittent inhalation-mouse TCLo; 10 ppm/6 hours/10 weeks intermittent inhalation-mouse TCLo; 302 ppm/6 hours/26 weeks intermittent inhalation-mouse TCLo; 4680 ppm/8 hours/4 days intermittent inhalation-mouse TCLo; 146,000 mg/m3 inhalation-dog LCLo; 170,000 mg/m3 inhalation-cat LCLo; 45,000 ppm/30 minutes inhalation-rabbit LCLo; >8263 mg/kg skin-rabbit LD50; >8263 mg/kg skin-guinea pig LD50; 50 mg/kg oral-man LDLo; 930 mg/kg oral-rat LD50; 6600 mg/kg/27 weeks intermittent oral-rat TDLo; 4700 mg/kg oral-mouse LD50; 2000 mg/kg oral-dog LDLo; 88 mg/kg intravenous-rabbit LDLo;

2890 ug/kg intraperitoneal-rat LD50; 340 mg/kg intraperitoneal-mouse LD50; 527 mg/kg intraperitoneal-guinea pig LDLo; 1500 mg/kg intraperitoneal-mammal LDLo; 1400 mg/kg subcutaneous-frog LDLo; 194 mg/kg unreported-man LDLo; mutagenic data (RTECS); reproductive effects data (RTECS); tumorigenic data (RTECS).

CARCINOGEN STATUS: OSHA Carcinogen; Known Human Carcinogen (NTP); Human Sufficient Evidence, Animal Sufficient Evidence (IARC Group-1). Numerous case reports and series have suggested a relationship between exposure to benzene and the occurrence of various types of leukemia. Several case-control studies have also shown increased odds ratios for exposure to benzene, but mixed exposure patterns and poorly defined exposures render their interpretation difficult. Three independent cohort studies have demonstrated an increased incidence of acute nonlymphocytic leukemia in workers exposed to benzene.

LOCAL EFFECTS: Irritant- inhalation, skin and eye.

ACUTE TOXICITY LEVEL: Moderately toxic by inhalation and ingestion; slightly toxic by dermal absorption.

TARGET EFFECTS: Central nervous system depressant; bone marrow depressant. Poisoning may also affect the immune system and the heart.

AT INCREASED RISK FROM EXPOSURE: Persons with certain immunological tendencies, poor nutrition, anemia and drug or chemically induced agranulocytemia.

ADDITIONAL DATA: Use of alcoholic beverages may enhance the toxic effects. Use of stimulants such as epinephrine may cause cardiac arrhythmias. May cross the placenta. Interactions with medications have been reported.

ALTH EFFECTS
LATION:

G. OLINE, AUTOMOTIVE, UNLEADED:

IRRITANT/NARCOTIC/ASPHYXIANT/CARCINOGEN.

ACUTE EXPOSURE- At 160-270 ppm throat irritation may occur within several hours. At 2000 ppm mild anesthesia may occur within 30 minutes. Other symptoms of central nervous system depression may include headache, nausea, vomiting, dizziness, drowsiness, facial flushing, blurred vision, slurred speech, difficulty swallowing, staggering, confusion and euphoria. At higher levels dyspnea, pulmonary edema and bronchopneumonia may develop. Further depression may occur with weak respiration and pulse, nervousness, twitching, irritability, and ataxia. Severe intoxication may result in delirium, unconsciousness, coma, and convulsions with epileptiform seizures. The pupils may be constricted or, in comatose states, fixed and dilated or unequal; nystagmus may also occur. May also affect the liver, kidneys, spleen, brain, myocardium and pancreas. Death may be due to respiratory or circulatory failure or ventricular fibrillation. Extremely high concentration may cause asphyxiation.

CHRONIC EXPOSURE- With few exceptions, most of the reported effects of repeated inhalation are from intentional "sniffing" of gasoline rather than workplace exposure. Reported symptoms include headache, nausea, fatigue, anorexia and weight loss, pallor, dizziness, insomnia, memory loss, nervousness, confusion, muscular weakness and cramps, peripheral neuropathy, polyneuritis, and neurasthenia. It is unclear whether some of these symptoms may have been due to gasoline containing lead. Liver and and kidney damage are also possible. In a 90 day study, male but not female rats exhibited a severe, dose-related renal toxicity. In another study, an increase in renal adenomas and carcinomas in male rats and an

increase in hepatocellular adenomas and carcinomas in female mice were reported.

BENZENE:
IRRITANT/NARCOTIC/BONE MARROW DEPRESSANT/CARCINOGEN.

ACUTE EXPOSURE- Concentrations of 3000 ppm may cause respiratory tract irritation; more severe exposures may result in pulmonary edema. Systemic effects are mainly on the central nervous system and depend on exposure time and concentration. No effects were noted at 25 ppm for 8 hours; signs of intoxication began at 50-150 ppm within 5 hours; at 500-1500 ppm, within 1 hour; were severe at 7500 ppm, within 30-60 minutes; and 20,000 ppm was fatal within 5-10 minutes. Effects may include nausea, vomiting, headache, dizziness, drowsiness, weakness, sometimes preceded by a brief period of exhilaration or euphoria, irritability, malaise, confusion, ataxia, staggering, weak, rapid pulse, chest pain and tightness with breathlessness, pallor, cyanosis of the lips and fingertips, and tinnitus. In severe exposures there may be blurred vision, shallow, rapid breathing, delirium, cardiac arrhythmias, unconsciousness, deep anesthesia, paralysis, and coma characterized by motor restlessness, tremors and hyperreflexia, sometimes preceded by convulsions. Recovery depends on the severity of exposure. Polyneuritis may occur and there may be persistent nausea, anorexia, muscular weakness, headache, drowsiness, insomnia, and agitation. Nervous irritability, breathlessness, and unsteady gait may persist for 2-3 weeks; a peculiar skin color and cardiac distress may persist for 4 weeks. Liver and kidney effects may occur, but are usually mild, temporary impairments. Chromosomal damage has been found after exposure to toxic levels. Although generally hematotoxicity is not a significant concern in acute exposure, delayed hematological effects, including anemia and thrombocytopenia, have been reported, as have petechial hemorrhages, spontaneous internal bleeding and secondary infections. In fatal exposures, death may be due to asphyxia, central nervous system depression, cardiac or respiratory failure and circulatory collapse, or occasionally, sudden ventricular fibrillation. It may occur within a few minutes to several hours, or cardiac arrythmia may occur at anytime within 24 hours. Also, death from central nervous system, respiratory or hemorrhagic complications may occur up to 5 days after exposure. Pathologic findings have included respiratory inflammation with edema and hemorrhage of the lungs, renal congestion, cerebral edema, and extensive petechial hemorrhages in the brain, pleurae, pericardium, urinary tract, mucous membranes, and skin.

CHRONIC EXPOSURE- Longterm exposure may cause symptoms referable to the central nervous, hematopoietic and immune systems. Early effects are vague and varied and may include headache, light-headedness, dizziness, nausea, anorexia, abdominal discomfort, and fatigue. Sore, dry throat, weakness, lethargy, malaise, drowsiness, nervousness, and irritability have also been reported. Later there may be dyspnea, pallor, slightly increased temperature, decreased blood pressure, rapid pulse, palpitations, and visual disturbances. Dizziness when cold water is placed in the ear and hearing impairment have been reported, as have diffuse cerebral atrophy associated with ataxia, tremors and emotional lability. Workers exposed to benzene in combination with other solvents have exhibited polyneuritis. Several case reports, one of them an acute exposure, suggest the possibility that systemic exposure may be associated with retrobulbar or optic neuritis. Occasionally hemorrhages in retina and conjunctiva

occur and rarely neuroretinal edema and papilledema have accompanied the retinal hemorrhages. Hematological effects vary widely and may appear after a few weeks or many years of exposure or even many years after exposure has ceased. The degree of exposure below which no blood effects will occur cannot be established with certainty. In the early stages, there may be blood clotting defects due to morphological, functional and quantitative platelet alteration with resultant bleeding from the nose and gums, easy bruising and petechiae; leukopenia with predominant lymphocytopenia or neutropenia; and anemia which may be normochromic or macrocytic and hypochromic. Extramedullary hematopoiesis, splenomegaly, circulating immature marrow cells, and an initial increase in leukocytes, erythrocytes and platelets have also been reported. The bone marrow may be hyper-, hypo- or normoplastic and does not always correlate with the peripheral blood picture. Also, the symptoms do not always parallel the laboratory findings. If treated at this stage, the effects appear reversible, although recovery may be protracted and there may be relapses. Decreased erythrocyte survival, hemolysis, capillary fragility, internal hemorrhages, iron metabolism disturbances, and hyperbilirubinemia have also been reported. Exposure to high levels for longer periods may result in aplasia and fatty degeneration of the bone marrow with pancytopenia. The most serious cases of aplastic anemia may be fatal due to hemorrhage and infection; death may occur within 3 months of diagnosis. Enormous variability in individual response, including non-dose dependent aplasia, and the finding of eosinophilia suggests that, in some cases, the blood dyscrasia may partially be an allergic reaction. Numerous case reports and series have suggested a relationship between exposure to benzene and the occurrence of various types of leukemia. Several case-control studies have also shown increased odds ratios for exposure to benzene, but mixed exposure patterns and poorly defined exposures render their interpretation difficult. Three independent cohort studies have demonstrated an increased incidence of acute nonlymphocytic leukemia in workers exposed to benzene. Several studies have also suggested a link between occupational exposure and multiple myeloma and lymphoma, both Hodgkin's and nonhodgkin's. Although aplastic anemia is probably the more likely consequence of longterm exposure, it is not uncommon for an individual surviving this, to go through a preleukemic phase into frank leukemia. Conversely, leukemia without precedent aplastic anemia can occur. In one study the range of time from the start of the exposure to the diagnosis of leukemia was 3-24 years. It has been suggested that the chromosomal aberrations which can arise in peripheral blood and bone marrow cells and persist for a long time after exposure ceases, may be associated with the increased incidence of leukemia. The immunosuppressive effect has also been suggested as being associated with the leukemogenesis. Adverse effects on the immunological system have been shown to make rabbits more susceptible to tuberculosis and pneumonia and may explain why the terminal event in some cases of benzene intoxication may be overwhelming infection. Exposed mice exhibited a tendency toward induction of lymphoid neoplasms. Rats exhibited an increased incidence of neoplasms, mainly carcinomas, at various sites. Menstrual disturbances have been reported more frequently in exposed women. Testicular damage has been reported in rats, rabbits and guinea pigs. Some animal studies have demonstrated embryo/fetotoxicity, sometimes at levels as low as 10 ppm and the potential for teratogenic effects such as decreased body weight and

skeletal variants, have also been shown. Other studies have not produced any abnormalities or embryolethality.

S[illegible] CONTACT:
GASOLINE, AUTOMOTIVE, UNLEADED:
IRRITANT.
ACUTE EXPOSURE- Liquid may cause irritation with erythema and pain. Prolonged or extensive contact may cause blistering and, in extreme cases epidermal necrolysis. A 12 year old boy partially immersed in a pool of gasoline for 1 hour experienced hypotension, abdominal tenderness, disseminated intravascular coagulation, transient hematuria, nonoliguric renal failure and an elevated serum amylase. Autopsy revealed cerebral edema, diffuse bilateral pneumonia, biventricular cardiac enlargement, toxic nephrosis, fatty infiltration of liver and peripancreatic fat necrosis.
CHRONIC EXPOSURE- Repeated or prolonged contact with the liquid may cause irritation, dermatitis and defatting of the skin with drying and cracking or burns and blistering. Some individuals may develop hypersensitivity, probably due to additives.

BENZENE:
IRRITANT.
ACUTE EXPOSURE- Direct contact may cause irritation. Effects may include erythema, a burning sensation, and with prolonged contact, blistering and edema. Under normal conditions, significant signs of systemic toxicity are unlikely from skin contact alone due to the slow rate of absorption; it may however, contribute to the toxicity from inhalation. Application to guinea pigs resulted in increased dermal permeability.
[illegible]ONIC EXPOSURE- Repeated or prolonged contact defats the skin and may result in dermatitis with erythema, scaling, dryness, vesiculation, and fissuring, possibly accompanied by paresthesias of the fingers which may persist several weeks after the dermatitis subsides. Peripheral neuritis has also been reported. Secondary infections may occur. Tests on guinea pigs indicate sensitization is possible. Although animal studies have failed to establish a relationship between skin contact and a carcinogenic effect, most of the studies were inadequate; some papillomas and hematopoietic effects have been reported.

EYE CONTACT:
GASOLINE, AUTOMOTIVE, UNLEADED:
IRRITANT.
ACUTE EXPOSURE- Concentrations between 270 and 900 ppm may cause a sensation of irritation often before signs such as conjunctival hyperemia are visible. Liquid splashed in the eyes may cause pain, smarting and slight, transient corneal epithelial disturbance. Blepharospasm and conjunctival hyperemia and edema may occur.
CHRONIC EXPOSURE- Repeated or prolonged exposure may cause conjunctivitis and possible gradual, irreversible loss of corneal and conjunctival sensitivity.

BENZENE:
IRRITANT.
ACUTE EXPOSURE- May cause irritation. Vapor concentrations of 3000 ppm are [illegible]ery irritating, even on brief exposure. Droplets cause a moderate

burning sensation, but only a slight, transient corneal epithelial injury with rapid recovery.
RONIC EXPOSURE- Repeated or prolonged exposure may cause conjunctivitis. 50% of rats exposed to 50 ppm for more than 600 hours developed cataracts.

NGESTION:
ASOLINE, AUTOMOTIVE, UNLEADED:
ARCOTIC.
ACUTE EXPOSURE- May cause irritation and burning of the gastrointestinal tract with nausea, vomiting and diarrhea. Absorption may cause initial central nervous stimulation followed by depression. Symptoms may include a mild excitation, restlessness, nervousness, irritability, twitching, weakness, blurred vision, headache, dizziness, drowsiness, incoordination, confusion, delirium, unconsciousness, convulsions and coma. Cardiac arrythmias may occur. Transient liver damage is possible. Direct or indirect aspiration may cause chemical pneumonitis with pulmonary edema and hemorrhage, possibly complicated by bacterial pneumonia, and less frequently, by emphysema and pneumonthorax. Signs of pulmonary involvement may include coughing, dyspnea, substernal pain, sudden development of rapid breathing, cyanosis, tachycardia and fever. Even small amounts may be fatal with death caused by cardiac arrest, asphyxia or respiratory paralysis. Depending on amount aspirated, death may occur rapidly or within 24 hours.
CHRONIC EXPOSURE- No data available.

ENZENE:
COTIC/CARCINOGEN.
TE EXPOSURE- May cause local irritation and burning sensation in the mouth, throat and stomach, and hemorrhagic inflammatory lesions of the mucous membranes in contact with the liquid. Signs and symptoms of systemic intoxication may include nausea, vomiting, headache, dizziness, weakness, staggering, chest pain and tightness, shallow, rapid pulse and respiration, breathlessness, pallor followed by flushing, and a fear of impending death. There may be visual disturbances, tremors, convulsions, ventricullar irregularities, and paralysis. Excitement, euphoria or delirium may precede weariness, fatigue, sleepiness and followed by stupor and unconsciousness, coma and death from respiratory failure. Those who survive the central nervous system effects may develop bronchitis, pneumonia, pulmonary edema, and intrapulmonary hemorrhage. Aspiration may cause immediate pulmonary edema and hemorrhage. The usual lethal dose in humans is 10-15 milliliters, but smaller amounts have been reported to cause death. A single exposure may produce longterm effects with pancytopenia persisting up to a year.
CHRONIC EXPOSURE- Daily administration to humans of 2-5 grams in olive oil caused headache, vertigo, bladder irritability, impotence, gastric disturbances, and evidence of renal congestion. In female rats treated with 132 single daily doses over 187 days, no effects were observed at 1 mg/kg; slight leukopenia at 10 mg/kg; and both leukopenia and anemia at 50 and 100 mg/kg. Oral administration to rats and mice at various dose levels induced neoplasms at multiple sites in males and females. In a one year gavage study, rats given 50 or 250 mg/kg, 4-5 days/week for 52 weeks did not exhibit acute or subacute toxic effects, but a dose correlated ncrease of leukemias and mammary carcinomas was observed; some other umor types were also reported. Reproductive effects have been reported

in animals.

SECTION 12 ECOLOGICAL INFORMATION

ENVIRONMENTAL IMPACT RATING (0-4): no data available

ACUTE AQUATIC TOXICITY: no data available

DEGRADABILITY: no data available

LOG BIOCONCENTRATION FACTOR (BCF): no data available

LOG OCTANOL/WATER PARTITION COEFFICIENT: no data available

SECTION 13 DISPOSAL INFORMATION

Observe all federal, state and local regulations when disposing of this substance.

Disposal must be in accordance with standards applicable to generators of hazardous waste, 40 CFR 262. EPA Hazardous Waste Number D001.
100 pound CERCLA Section 103 Reportable Quantity.

Benzene - Regulatory level: 0.5 mg/l (TCLP-40 CFR 261 Appendix II)
Materials which contain the above substance at or above the TCLP regulatory level meet the EPA toxicity characteristic, and must be disposed of in accordance with 40 CFR part 262. EPA Hazardous Waste Number D018.

SECTION 14 TRANSPORTATION INFORMATION

U.S. DEPARTMENT OF TRANSPORTATION SHIPPING NAME-ID NUMBER, 49 CFR 172.101:
Gasoline-UN 1203

U.S. DEPARTMENT OF TRANSPORTATION HAZARD CLASS OR DIVISION, 49 CFR 172.101:
3 - Flammable liquid

U.S. DEPARTMENT OF TRANSPORTATION PACKING GROUP, 49 CFR 172.101:
PG II

U.S. DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS, 49 CFR 172.101
AND SUBPART E:
Flammable liquid

U.S. DEPARTMENT OF TRANSPORTATION PACKAGING AUTHORIZATIONS:
EXCEPTIONS: 49 CFR 173.150
NON-BULK PACKAGING: 49 CFR 173.202
BULK PACKAGING: 49 CFR 173.242

U.S. DEPARTMENT OF TRANSPORTATION QUANTITY LIMITATIONS 49 CFR 172.101:
PASSENGER AIRCRAFT OR RAILCAR: 5 l
CARGO AIRCRAFT ONLY: 60 l

CTION 15 REGULATORY INFORMATION

SCA STATUS: Y

ERCLA SECTION 103 (40CFR302.4): Y 10 pounds RQ
ARA SECTION 302 (40CFR355.30): N
ARA SECTION 304 (40CFR355.40): N
ARA SECTION 313 (40CFR372.65): Y
SHA PROCESS SAFETY (29CFR1910.119): N
ALIFORNIA PROPOSITION 65: Y

ARA HAZARD CATEGORIES, SARA SECTIONS 311/312 (40 CFR 370.21)
CUTE HAZARD: Y
HRONIC HAZARD: Y
IRE HAZARD: Y
EACTIVITY HAZARD: N
UDDEN RELEASE HAZARD: N

SECTION 16 OTHER

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MATERIAL SAFETY DATA SHEET

Date Issued: 02/13/96

SECTION A - IDENTIFICATION & EMERGENCY INFORMATION

Manufacturer's Name: Dryden Oil Company, Inc.
Address: 9300 Pulaski Highway
Baltimore, MD 21220

Emergency Telephone Number: 410-574-5000
800-777-1466

PRODUCT NAME: Drydene H.D. Lithium E.P. Grease 0, 1 and 2

Chemical Name: Petroleum Oil (Grease)

Product Appearance & Odor: Brown Semi-Solid
Bland Odor

CAS Number (For Finished Product):
COMPLEX MIXTURE
CAS Number Not Applicable

HAZARDOUS MATERIALS IDENTIFICATION SYSTEM (HMIS)
Health - 1 Flammability - 1 Reactivity - 0
Hazard Rating: Lease-0 Slight-1 Moderate-2 High-3 Extreme-4

SECTION B - COMPONENTS & HAZARD INFORMATION

COMPONENTS	CAS NO. OF COMPONENTS	APPROXIMATE CONCENTRATION
Lubricating Oil Base Stock	64742-65-0	Greater than 85%
Proprietary Additives	Mixture	Less than 15%

Exposure Limit for Total Product: 5mg/m³ oil mist for an 8-hour work day. Basis: OSHA Reg 29 CFR 1910.1000
CERCLA Hazardous Substances: None known. If this product is accidentally spilled, it is not subject to any special reporting under the requirements of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). We recommend you contact local authorities to determine if there may be other local reporting requirements. US TSCA Inventory: All components of this material are on the US TSCA Inventory. Threshold Planning Quantity (TPQ), EPA Regulation 40 CFR 355 Extremely Hazardous Substances (SARA Sections 301-304): None. Toxic Chemical Release Reporting, EPA Regulation 40 CFR 372 (SARA Section 313):
Zinc Alkyldithiophosphate CAS 68649-42-3 1% max

SECTION C - PHYSICAL DATA (The following data are approximate or typical values.)

Boiling Range: Not Determined

Percent Volatile by Volume: NEGLIGIBLE

Specific Gravity (H_2O=1): .87/.90 @ 16.C

Vapor Pressure: NEGLIGIBLE

Pour Point: Freezing Point 0.F

Vapor Density: GREATER THAN AIR

Viscosity: N/A

Evaporation Rate: NEGLIGIBLE

Solubility in Water: Negligible, Below 0.1%

SECTION D - FIRE PROTECTION INFORMATION

FLASH POINT & METHOD: Min. ASTM D-92 C.O.C. °C, (°F.)
400. F

AUTOIGNITION TEMPERATURE:
NOT DETERMINED

NATIONAL FIRE
PROTECTION ASSOCIATION
(NFPA)-Hazard Identification
Health - 1
Flammability - 1
Reactivity - 0
Basis: Recommended by Dryden Oil Co.
Hazard Rating (NFPA):
4-Extreme 3-High 2-Moderate
1-Slight 0-Insignificant

UNUSUAL FIRE & EXPLOSION HAZARDS:
None

Flammability Limits (% by volume in air):
Lower: Not determined Upper: Not determined

SECTION D - FIRE PROTECTION INFORMATION (Continued)

HANDLING PRECAUTIONS: Use product with caution around heat, sparks, pilot lights, static electricity and open flame.

DECOMPOSITION PRODUCTS UNDER FIRE CONDITIONS: Fumes, smoke, carbon monoxide, sulfur oxides, and other decomposition products, in the case of incomplete combustion.

EXTINGUISHING MEDIA & FIRE FIGHTING PROCEDURES: Foam, water spray (fog), dry chemical, carbon dioxide and vaporizing liquid type extinguishing agents may all be suitable for extinguishing fires involving this type of product, depending on the size or potential size of fire and circumstances related to the situation. Plant fire protection and response strategy through consultation with local fire protection authorities or appropriate specialists.

The following procedures for this type of product are based on the recommendations in the National Fire Protection Associations' *Fire Protection Guide on Hazardous Materials*. Use water spray, dry chemical, foam, or carbon dioxide to extinguish the fire. Water or foam may cause frothing. Use water to keep fire-exposed containers cool. Water spray may be used to flush spills away from exposures. Minimize breathing of gases, vapor, fumes or decomposition products. Use supplied-air breathing equipment for enclosed or confined spaces or as otherwise needed.

EMPTY CONTAINER WARNING: "Empty" containers retain residue (liquid and/or vapor) and can be dangerous. DO NOT PRESSURIZE, CUT, WELD, BRAZE, SOLDER, DRILL, GRIND OR EXPOSE SUCH CONTAINERS TO HEAT, FLAME, SPARKS OR OTHER SOURCES OF IGNITION: THEY MAY EXPLODE AND CAUSE INJURY OR DEATH. Do not attempt to clean since residue is difficult to remove. "Empty" drums should be completely drained, properly bunged and promptly returned to a drum reconditioner. All other containers should be disposed of in an environmentally safe manner and in accordance with governmental regulations.

SECTION E - PROTECTION & PRECAUTIONS

VENTILATION: Use local exhaust to capture vapor, mists or fumes, if necessary. Provide ventilation sufficient to prevent exceeding recommended exposure limit or buildup of explosive concentrations of vapor in air. No smoking, flame or other ignition sources.

RESPIRATORY PROTECTION: Use supplied-air respiratory protection in confined or enclosed spaces, if needed.

PROTECTIVE GLOVES: Use chemical-resistant gloves, if needed, to avoid prolonged or repeated skin contact.

EYE PROTECTION: Use splash goggles or face shield when eye contact may occur.

OTHER PROTECTIVE EQUIPMENT: Use chemical-resistant apron or other impervious clothing, if needed, to avoid contaminating regular clothing, which could result in prolonged or repeated skin contact.

WORK PRACTICES/ENGINEERING CONTROLS: Keep containers closed when not in use. Do not store near heat, sparks, flame or strong oxidants. In order to prevent fire or explosion hazards, use appropriate equipment.

PERSONAL HYGIENE: Minimize breathing vapor, mist or fumes. Avoid prolonged or repeated contact with skin. Remove contaminated clothing: launder or dry-clean before re-use. Remove contaminated shoes and thoroughly clean before re-use; discard if oil-soaked. Cleanse skin thoroughly after contact, before breaks and meals, and at end of work period. Product is readily removed from skin by waterless hand cleaners followed by washing thoroughly with soap and water.

VARIABILITY AMONG INDIVIDUALS: Health studies have shown that many petroleum hydrocarbons and synthetic lubricants pose potential human health risks which may vary from person to person. As a precaution, exposure to liquids, vapors, mists or fumes should be minimized.

SECTION F - SPILL OR LEAK PROCEDURE

ENVIRONMENTAL IMPACT: Report spills as required to the appropriate authorities. U.S. Coast Guard Regulations require immediate reporting of spills that could reach any waterway including intermittent dry creeks. Report spill to the Coast Guard toll free number 800-424-8802. **PROCEDURES IF MATERIAL IS RELEASED OR SPILLED:** Recover free product. Add sand, earth, or other suitable absorbent material to the spill area. Minimize breathing vapors. Minimize skin contact. Keep product out of sewers and watercourses by diking or impounding. Advise authorities if the product has entered or may enter sewers, watercourses, or extensive land areas.

ASSURE CONFORMITY WITH ALL APPLICABLE REGULATIONS.

WASTE DISPOSAL: Dispose of in an environmentally safe manner and in accordance with all government regulations to include Federal, State, and local requirements.

SECTION G - REACTIVITY

STABILITY: Stable

HAZARDOUS POLYMERIZATION: Will not occur.

CONDITIONS & MATERIALS TO AVOID: Avoid heat, open flames and oxidizing materials.

HAZARDOUS DECOMPOSITION PRODUCTS: Thermal decomposition products are highly dependent on the combustion conditions. A complex mixture of airborne solid, liquid, particulates and gases will evolve when this material undergoes combustion. Carbon monoxide and other unidentified organic compounds may be formed upon combustion.

SECTION H - EMERGENCY & FIRST AID PROCEDURES AND PRIMARY ROUTES OF ENTRY

EYE CONTACT: If splashed into the eyes, flush with clear water for 15 minutes or until irritation subsides. If irritations persists, call a physician. **SKIN CONTACT:** In case of skin contact, remove any contaminated clothing and wash skin thoroughly with soap and water. **INGESTION:** If ingested, DO NOT induce vomiting; call a physician immediately.

INHALATION: Vapor pressure is very low. Vapor inhalation under ambient temperature conditions is not normally a problem. If overcome by vapor from hot product, immediately remove from exposure and call a physician. Administer oxygen, if available. If over-exposed to oil mist, remove from further exposure until excessive mist oil condition subsides.

SECTION I - EFFECTS OF OVEREXPOSURE

SKIN: Prolonged or repeated skin contact may cause skin irritation. **EYE:** May cause eye irritation. **INGESTION:** Relatively non-toxic.

SECTION J - TRANSPORTATION INFORMATION

DEPARTMENT OF TRANSPORTATION (DOT) - DOT Identification Number: Not Regulated.

THE PRECISE COMPOSITION OF THIS MIXTURE IS PROPRIETARY INFORMATION. A MORE COMPLETE DISCLOSURE WILL BE PROVIDED TO A PHYSICIAN OR NURSE IN THE EVENT OF A MEDICAL EMERGENCY.

'6274

CTION 1 CHEMICAL PRODUCTS & COMPANY IDENTIFICATION

OCCUPATIONAL HEALTH SERVICES, INC.
11 WEST 42ND STREET, 12TH FLOOR
NEW YORK, NEW YORK 10036
1-800-445-MSDS (1-800-445-6737) OR
1-212-789-3535

FOR EMERGENCY SOURCE INFORMATION
CONTACT: 1-615-366-2000 USA

SUBSTANCE: DIESEL FUEL NO. 4

TRADE NAMES/SYNONYMS:
MARINE DIESEL FUEL; DISTILLATE MARINE DIESEL FUEL; NAVAL DISTILLATE FUEL; MARINE DISTILLATE OIL; MDO; OHS76274

CHEMICAL FAMILY:
Petroleum hydrocarbon

CREATION DATE: 09/26/91 REVISION DATE: 01/15/94

SECTION 2 COMPOSITION/INFORMATION ON INGREDIENTS

COMPONENT : DIESEL FUEL NO. 4
PERCENTAGE: >99.0

R CONTAMINANTS: MAY CONTAIN TRACES OF SULFUR

SECTION 3 HAZARDS IDENTIFICATION

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=2 REACTIVITY=0 PERSISTENCE=1
NFPA RATINGS (SCALE 0-4): HEALTH=U FIRE=2 REACTIVITY=0

EMERGENCY OVERVIEW:
Pale yellow liquid.
Suspect cancer hazard (contains material which can cause cancer in animals). Risk of cancer depends on duration and level of contact. May be irritating to the respiratory tract and eyes. May affect the central nervous system. May damage the lungs. Combustible liquid and vapor.
Keep away from all ignition sources. Avoid breathing vapor or mist. Avoid contact with eyes, skin and clothing. Keep container tightly closed. Wash thoroughly after handling. Use only with adequate ventilation.

POTENTIAL HEALTH EFFECTS:
INHALATION:
SHORT TERM EFFECTS: May cause irritation. Additional effects may include coughing, lack of appetite, nausea, vomiting, difficulty breathing, headache, weakness, dizziness, confusion, disorientation, incoordination, restlessness, excitation, bluish skin color, unconsciousness and coma.
ONG TERM EFFECTS: No information available on significant adverse effects.
CONTACT:

SHORT TERM EFFECTS: No information is available.
LONG TERM EFFECTS: No information is available.
CONTACT:
SHORT TERM EFFECTS: May cause irritation.
LONG TERM EFFECTS: No information available on significant adverse effects.
INGESTION:
SHORT TERM EFFECTS: May cause coughing, nausea, vomiting, diarrhea, difficulty breathing, drunkeness and lung damage.
LONG TERM EFFECTS: No information is available.
ADDITIONAL DATA: May cause cancer.

CARCINOGEN STATUS:
OSHA: N
NTP: N
IARC: Y

SECTION 4 FIRST AID MEASURES

INHALATION:
FIRST AID- Remove from exposure area to fresh air immediately. If breathing has stopped, perform artificial respiration. Keep person warm and at rest. Treat symptomatically and supportively. Get medical attention immediately.

SKIN CONTACT:
data available

EYE CONTACT:
FIRST AID- Wash eyes immediately with large amounts of water or normal saline, occasionally lifting upper and lower lids, until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

INGESTION:
FIRST AID- Only hydrocarbons that are solvents for a toxic agent or are themselves toxic need be evacuated. Extreme care must be used to prevent aspiration. Gastric lavage with a cuffed endotracheal tube in place to prevent further aspiration should be done within 15 minutes. In the absence of depression or convulsions or impaired gag reflex, emesis can also be induced using syrup of ipecac without increasing the hazard of aspiration. Treat symptomatically and supportively. Gastric lavage should be performed by qualified medical personnel. Get medical attention immediately.

NOTE TO PHYSICIAN
ANTIDOTE:
No specific antidote. Treat symptomatically and supportively.

SECTION 5 FIRE FIGHTING MEASURES

FIRE AND EXPLOSION HAZARD:
Moderate fire hazard when exposed to heat or flame.

rs are heavier than air and may travel a considerable distance to a sour

of ignition and flash back.

.[illegible]r-air mixtures are explosive above flash point.

EXTINGUISHING MEDIA:
Dry chemical, carbon dioxide, water spray or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

For larger fires, use water spray, fog or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

FIREFIGHTING:
Move container from fire area if you can do it without risk. Apply cooling water to sides of containers that are exposed to flames until well after fire is out. Stay away from ends of tanks. For massive fire in cargo area, use unmanned hose holder or monitor nozzles; if this is impossible, withdraw from area and let fire burn. Withdraw immediately in case of rising sound from venting safety device or any discoloration of tank due to fire. Isolate for 1/2 mile in all directions if tank, rail car or tank truck is involved in fire (1990 Emergency Response Guidebook, DOT P 5800.5, Guide Page 27).

Extinguish only if flow can be stopped; use flooding amounts of water as a fog, solid streams may be ineffective. Cool containers with flooding amounts of water, apply from as far a distance as possible. Avoid breathing vapors, keep upwind.

FLASH POINT: 140 F (60 C) (PMCC)
FLAMMABILITY CLASS(OSHA): IIIA

HAZARDOUS COMBUSTION PRODUCTS:
Thermal decomposition products may include toxic oxides of sulfur and carbon.

SECTION 6 ACCIDENTAL RELEASE MEASURES

OCCUPATIONAL SPILL:
Shut off ignition sources. Stop leak if you can do it without risk. Use water spray to reduce vapors. For small spills, take up with sand or other absorbent material and place into containers for later disposal. For larger spills, dike far ahead of spill for later disposal. No smoking, flames or flares in hazard area. Keep unnecessary people away; isolate hazard area and restrict entry.

SECTION 7 HANDLING AND STORAGE

Observe all federal, state and local regulations when storing this substance.

Store in accordance with 29 CFR 1910.106.

Bonding and grounding: Substances with low electroconductivity, which may be ignited by electrostatic sparks, should be stored in containers which meet the bonding and grounding guidelines specified in NFPA 77-1983, Recommended Practice on Static Electricity.

Store away from incompatible substances.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

EXPOSURE LIMITS:
MINERAL OIL MIST:
5 mg/m3 OSHA TWA
5 mg/m3 ACGIH TWA; 10 mg/m3 ACGIH STEL
(Notice of Intended Changes 1993-94)
5 mg/m3 NIOSH recommended TWA;
10 mg/m3 NIOSH recommended STEL

Measurement method: Particulate filter;
1,1,2-trichloro-1,2,2-trifluoroethane; infrared spectrometry;
(NIOSH Vol. III # 5026).

HYDROGEN SULFIDE:
10 ppm (14 mg/m3) OSHA TWA; 15 ppm (21 mg/m3) OSHA STEL
10 ppm (14 mg/m3) ACGIH TWA; 15 ppm (21 mg/m3) ACGIH STEL
10 ppm NIOSH recommended 10 minute ceiling
10 ppm (14 mg/m3) DFG MAK TWA;
20 ppm (28 mg/m3) DFG MAK 10 minute peak, momentary value; 4 times/shift

Measurement method: Drying tube/molecular sieve tube; thermal desorption apparatus; gas chromatography with flame ionization detection; (NIOSH Vol. II(6) # 296).

0 pounds SARA Section 302 Threshold Planning Quantity
100 pounds SARA Section 304 Reportable Quantity
100 pounds CERCLA Section 103 Reportable Quantity
1500 pounds OSHA Process Safety Management Threshold Quantity

OSHA revoked the final rule limits of January 19, 1989 in response to the 11th Circuit Court of Appeals decision (AFL-CIO v. OSHA) effective June 30, 1993. See 29 CFR 1910.1000 (58 FR 35338)

VENTILATION:
Process enclosure ventilation recommended to meet published exposure limits. Ventilation equipment must be explosion-proof.

EYE PROTECTION:
Employee must wear splash-proof or dust-resistant safety goggles and a faceshield to prevent contact with this substance.

Emergency wash facilities:
Where there is any possibility that an employee's eyes and/or skin may be exposed to this substance, the employer should provide an eye wash fountain and quick drench shower within the immediate work area for emergency use.

CLOTHING:
Wear oil impervious clothing. Avoid prolonged or repeated contact with substance. Avoid wearing oil soaked clothing.

GLOVES:
ployee must wear appropriate protective gloves to prevent contact with this tance.

RESPIRATOR:
The following respirators are recommended based on information found in the physical data, toxicity and health effects sections. They are ranked in order from minimum to maximum respiratory protection.
The specific respirator selected must be based on contamination levels found in the work place, must be based on the specific operation, must not exceed the working limits of the respirator and must be jointly approved by the National Institute for Occupational Safety and Health and the Mine Safety and Health Administration (NIOSH-MSHA).

Any type 'C' supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure mode or with a full facepiece, helmet or hood operated in continuous-flow mode.

Any self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.

FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode.

ny supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

DESCRIPTION: Pale yellow liquid.
BOILING POINT: 272-725 F (133-385 C)
SPECIFIC GRAVITY: 0.840-0.898 @ 60 F
WATER SOLUBILITY: insoluble
VISCOSITY: 1.7-4.3 cSt @ 140 F

SECTION 10 STABILITY AND REACTIVITY

REACTIVITY:
Stable under normal temperatures and pressures.

CONDITIONS TO AVOID:
Avoid contact with heat, sparks, flames, or other sources of ignition. Vapors may be explosive. Avoid overheating of containers; containers may violently rupture in heat of fire. Avoid contamination of water sources.

ace amounts of hydrogen sulfide may be present in this product. There is tential for accumulation of hydrogen sulfide in the head space of

containers or in enclosed areas where the product is stored, handled or used.

COMPATIBILITIES:
DIESEL FUEL NO. 4:
OXIDIZERS (STRONG): Fire and exposion hazard.

HAZARDOUS DECOMPOSITION:
Thermal decomposition products may include toxic oxides of sulfur and carbon.

POLYMERIZATION:
Hazardous polymerization has not been reported to occur under normal temperatures and pressures.

SECTION 11 TOXICOLOGY INFORMATION

DIESEL FUEL NO. 4:
CARCINOGEN STATUS: Animal Limited Evidence (IARC Group-2B). Skin application induced squamous-cell tumors in mice.
ACUTE TOXICITY LEVEL: No data available.
TARGET EFFECTS: Poisoning may affect the liver, kidneys, and lung.*

* Based on general information on diesel fuels.

HEALTH EFFECTS
INHALATION:
DIESEL FUEL NO. 4:
ACUTE EXPOSURE- Diesel fuel vapors or mist may cause respiratory tract irritation. A human exposure to diesel fuel has resulted in immediate cough, dyspnea, cyanosis and unconsciousness for one hour. A productive cough with sputum smelling of diesel fuel persisted for 37 days. Chest X-rays showed diffuse shadowing, most prominent at the lung bases, which resolved slowly with treatment but was still present at day 37. High levels may also cause central nervous system excitation followed by depression with symptoms possibly including restlessness, confusion, ataxia, headache, dizziness, anorexia, nausea, vomiting, weakness, incoordination, stupor, delirium and coma.
CHRONIC EXPOSURE- Prolonged or repeated exposure to diesel fuel vapors may cause irritation.

SKIN CONTACT:
FIRST AID- Remove contaminated clothing and shoes immediately. Wash affected area with soap or mild detergent and large amounts of water until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

EYE CONTACT:
DIESEL FUEL NO. 4:
ACUTE EXPOSURE- Liquid or vapor may cause slight irritation, although tests with one sample of diesel fuel in rabbit eyes was non-irritating.
CHRONIC EXPOSURE- Repeated or prolonged exposure may cause irritation.

INGESTION:
DIESEL FUEL NO. 4:

ACUTE EXPOSURE- Diesel fuel may cause nausea, vomiting, cramping, diarrhea, and possibly symptoms of central nervous system depression. Aspiration of even small amounts during ingestion or vomiting may result in severe pulmonary irritation with coughing, gagging, dyspnea, substernal distress, pneumonitis, pulmonary edema and hemorrhage, and death.
CHRONIC EXPOSURE- No data available.

SECTION 12 ECOLOGICAL INFORMATION

ENVIRONMENTAL IMPACT RATING (0-4): no data available

ACUTE AQUATIC TOXICITY: no data available

DEGRADABILITY: no data available

LOG BIOCONCENTRATION FACTOR (BCF): no data available

LOG OCTANOL/WATER PARTITION COEFFICIENT: no data available

SECTION 13 DISPOSAL INFORMATION

Observe all federal, state and local regulations when disposing of this substance.

Disposal must be in accordance with standards applicable to generators of hazardous waste, 40 CFR 262. EPA Hazardous Waste Number D001.
pound CERCLA Section 103 Reportable Quantity.

SECTION 14 TRANSPORTATION INFORMATION

U.S. DEPARTMENT OF TRANSPORTATION SHIPPING NAME-ID NUMBER, 49 CFR 172.101:
Diesel fuel-NA 1993

U.S. DEPARTMENT OF TRANSPORTATION HAZARD CLASS OR DIVISION, 49 CFR 172.101:
3 - Flammable liquid

U.S. DEPARTMENT OF TRANSPORTATION PACKING GROUP, 49 CFR 172.101:
PG III

U.S. DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS, 49 CFR 172.101 AND SUBPART E:
None

U.S. DEPARTMENT OF TRANSPORTATION PACKAGING AUTHORIZATIONS:
EXCEPTIONS: 49 CFR 173.150
NON-BULK PACKAGING: 49 CFR 173.203
BULK PACKAGING: 49 CFR 173.241

U.S. DEPARTMENT OF TRANSPORTATION QUANTITY LIMITATIONS 49 CFR 172.101:
PASSENGER AIRCRAFT OR RAILCAR: 60 l
AIRCRAFT ONLY: 220 l

SECTION 15 REGULATORY INFORMATION

TSCA STATUS: N

CERCLA SECTION 103 (40CFR302.4):	Y	100	pounds RQ
SARA SECTION 302 (40CFR355.30):	Y	500	pounds TPQ
SARA SECTION 304 (40CFR355.40):	Y	100	pounds RQ
SARA SECTION 313 (40CFR372.65):	N		
OSHA PROCESS SAFETY (29CFR1910.119):	Y	1500	pounds TQ
CALIFORNIA PROPOSITION 65:	N		

SARA HAZARD CATEGORIES, SARA SECTIONS 311/312 (40 CFR 370.21)

ACUTE HAZARD:	N
CHRONIC HAZARD:	Y
FIRE HAZARD:	Y
REACTIVITY HAZARD:	N
SUDDEN RELEASE HAZARD:	N

SECTION 16 OTHER

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CTION 1 CHEMICAL PRODUCTS & COMPANY IDENTIFICATION

OCCUPATIONAL HEALTH SERVICES, INC.
11 WEST 42ND STREET, 12TH FLOOR
NEW YORK, NEW YORK 10036
1-800-445-MSDS (1-800-445-6737) OR
1-212-789-3535

FOR EMERGENCY SOURCE INFORMATION
CONTACT: 1-615-366-2000 USA

CAS NUMBER: 68476-34-6

SUBSTANCE: DIESEL FUEL NO. 2

TRADE NAMES/SYNONYMS:
DIESEL OIL; DIESEL FUEL; DIESEL OIL, MEDIUM; FUELS, DIESEL, NO. 2; DIESEL OIL NO. 2-D; DIESEL FUEL OIL NO. 2-D; DIESEL FUEL NO. 2-D; NO. 2 DIESEL FUEL; WINTER DIESEL; CHEVRON DIESEL FUEL NO. 2; ARCO DIESEL (ARCO PRODUCTS COMPANY); DIESEL FUEL #2; REGULAR DIESEL; FUEL OIL#2; OHS07100

CHEMICAL FAMILY:
Petroleum hydrocarbon

CREATION DATE: 03/14/85 REVISION DATE: 01/15/94

CTION 2 COMPOSITION/INFORMATION ON INGREDIENTS

COMPONENT : DIESEL FUEL NO. 2-D
CAS NUMBER: 68476-34-6
PERCENTAGE: >99

OTHER CONTAMINANTS: MAY CONTAIN TRACES OF SULFUR

SECTION 3 HAZARDS IDENTIFICATION

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=2 REACTIVITY=0 PERSISTENCE=1
NFPA RATINGS (SCALE 0-4): HEALTH=0 FIRE=2 REACTIVITY=0

EMERGENCY OVERVIEW:
Colorless to yellow-brown liquid with a mild petroleum odor.
Suspect cancer hazard (contains material which can cause cancer in animals). Risk of cancer depends on duration and level of contact. Causes respiratory tract and skin irritation. May be irritating to eyes. May affect the central nervous system. May affect the kidneys. May damage the lungs. Combustible liquid and vapor.
Keep away from all ignition sources. Avoid breathing vapor or mist. Avoid contact with eyes, skin and clothing. Keep container tightly closed. Wash thoroughly after handling. Use only with adequate ventilation.

TIAL HEALTH EFFECTS:

INHALATION:
SHORT TERM EFFECTS: May cause irritation. Additional effects may include coughing, lack of appetite, nausea, vomiting, headache, weakness, drunkeness, confusion, disorientation, restlessness, excitation, bluish skin color, unconsciousness and coma.
LONG TERM EFFECTS: No information available on significant adverse effects.
SKIN CONTACT:
SHORT TERM EFFECTS: May cause irritation. Additional effects may include blisters and sores.
LONG TERM EFFECTS: In addition to effects from short term exposure, lack of appetite and kidney damage may occur.
EYE CONTACT:
SHORT TERM EFFECTS: May cause irritation.
LONG TERM EFFECTS: No information available on significant adverse effects.
INGESTION:
SHORT TERM EFFECTS: May cause coughing, nausea, vomiting, diarrhea, difficulty breathing, drunkeness and lung damage.
LONG TERM EFFECTS: No information is available.
ADDITIONAL DATA: May cause cancer.

CARCINOGEN STATUS:
OSHA: N
NTP: N
IARC: Y

SECTION 4 FIRST AID MEASURES

INHALATION:
FIRST AID- Remove from exposure area to fresh air immediately. If breathing has stopped, perform artificial respiration. Keep person warm and at rest. Treat symptomatically and supportively. Get medical attention immediately.

SKIN CONTACT:
FIRST AID- Remove contaminated clothing and shoes immediately. Wash affected area with soap or mild detergent and large amounts of water until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

EYE CONTACT:
FIRST AID- Wash eyes immediately with large amounts of water or normal saline, occasionally lifting upper and lower lids, until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

INGESTION:
FIRST AID- Only hydrocarbons that are solvents for a toxic agent or are themselves toxic need be evacuated. Extreme care must be used to prevent aspiration. Gastric lavage with a cuffed endotracheal tube in place to prevent further aspiration should be done within 15 minutes. In the absence of depression or convulsions or impaired gag reflex, emesis can also be induced using syrup of ipecac without increasing the hazard of aspiration. Treat symptomatically and supportively. Gastric lavage should be performed by qualified medical personnel. Get medical attention immediately.

E TO PHYSICIAN
DOTE:
Specific antidote. Treat symptomatically and supportively.

SECTION 5 FIRE FIGHTING MEASURES

FIRE AND EXPLOSION HAZARD:
Moderate fire hazard when exposed to heat or flame.

Vapors are heavier than air and may travel a considerable distance to a source of ignition and flash back.

Vapor-air mixtures are explosive above flash point.

EXTINGUISHING MEDIA:
Dry chemical, carbon dioxide, water spray or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

For larger fires, use water spray, fog or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

FIREFIGHTING:
Move container from fire area if you can do it without risk. Apply cooling water to sides of containers that are exposed to flames until well after fire out. Stay away from ends of tanks. For massive fire in cargo area, use nned hose holder or monitor nozzles; if this is impossible, withdraw from a and let fire burn. Withdraw immediately in case of rising sound from venting safety device or any discoloration of tank due to fire. Isolate for 1/2 mile in all directions if tank, rail car or tank truck is involved in fire (1990 Emergency Response Guidebook, DOT P 5800.5, Guide Page 27).

Extinguish only if flow can be stopped; use flooding amounts of water as a fog, solid streams may be ineffective. Cool containers with flooding amounts of water, apply from as far a distance as possible. Avoid breathing vapors, keep upwind.

FLASH POINT: >125 F (>52 C)
LOWER FLAMMABLE LIMIT: 0.6%
UPPER FLAMMABLE LIMIT: 7.5%
AUTOIGNITION: >475 F (>246 C)
FLAMMABILITY CLASS(OSHA): II

HAZARDOUS COMBUSTION PRODUCTS:
Thermal decomposition products may include toxic oxides of sulfur and carbon.

SECTION 6 ACCIDENTAL RELEASE MEASURES

OCCUPATIONAL SPILL:
Shut off ignition sources. Stop leak if you can do it without risk. Use water y to reduce vapors. For small spills, take up with sand or other absorbent ial and place into containers for later disposal. For larger spills, dike

ar ahead of spill for later disposal. No smoking, flames or flares in hazard ea. Keep unnecessary people away; isolate hazard area and restrict entry.

SECTION 7 HANDLING AND STORAGE

Observe all federal, state and local regulations when storing this substance.

Store in accordance with 29 CFR 1910.106.

Bonding and grounding: Substances with low electroconductivity, which may be ignited by electrostatic sparks, should be stored in containers which meet the bonding and grounding guidelines specified in NFPA 77-1983, Recommended Practice on Static Electricity.

Store away from incompatible substances.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

EXPOSURE LIMITS:
MINERAL OIL MIST:
5 mg/m3 OSHA TWA
5 mg/m3 ACGIH TWA; 10 mg/m3 ACGIH STEL
(Notice of Intended Changes 1993-94)
5 mg/m3 NIOSH recommended TWA;
10 mg/m3 NIOSH recommended STEL

easurement method: Particulate filter;
1,1,2-trichloro-1,2,2-trifluoroethane; infrared spectrometry;
(NIOSH Vol. III # 5026).

HYDROGEN SULFIDE:
10 ppm (14 mg/m3) OSHA TWA; 15 ppm (21 mg/m3) OSHA STEL
10 ppm (14 mg/m3) ACGIH TWA; 15 ppm (21 mg/m3) ACGIH STEL
10 ppm NIOSH recommended 10 minute ceiling
10 ppm (14 mg/m3) DFG MAK TWA;
20 ppm (28 mg/m3) DFG MAK 10 minute peak, momentary value; 4 times/shift

Measurement method: Drying tube/molecular sieve tube; thermal desorption apparatus; gas chromatography with flame ionization detection; (NIOSH Vol. II(6) # 296).

500 pounds SARA Section 302 Threshold Planning Quantity
100 pounds SARA Section 304 Reportable Quantity
100 pounds CERCLA Section 103 Reportable Quantity
1500 pounds OSHA Process Safety Management Threshold Quantity

OSHA revoked the final rule limits of January 19, 1989 in response to the 11th Circuit Court of Appeals decision (AFL-CIO v. OSHA) effective June 30, 1993. See 29 CFR 1910.1000 (58 FR 35338)

TILATION:
ide local exhaust ventilation to meet published exposure limits.

Ventilation equipment should be explosion-proof if explosive concentrations of dust, vapor or fume are present.

EYE PROTECTION:
Employee must wear splash-proof or dust-resistant safety goggles to prevent eye contact with this substance.

Emergency eye wash: Where there is any possibility that an employee's eyes may be exposed to this substance, the employer should provide an eye wash fountain within the immediate work area for emergency use.

CLOTHING:
Wear oil impervious clothing. Avoid prolonged or repeated contact with substance. Avoid wearing oil soaked clothing.

GLOVES:
Employee must wear appropriate protective gloves to prevent contact with this substance.

RESPIRATOR:
The following respirators are recommended based on information found in the physical data, toxicity and health effects sections. They are ranked in order from minimum to maximum respiratory protection.
The specific respirator selected must be based on contamination levels found in the work place, must be based on the specific operation, must not exceed the working limits of the respirator and must be jointly approved by the National Institute for Occupational Safety and Health and the Mine Safety and Health Administration (NIOSH-MSHA).

Any type 'C' supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure mode or with a full facepiece, helmet or hood operated in continuous-flow mode.

Any self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.

FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode.

Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

DESCRIPTION: Colorless to yellow-brown liquid with a mild petroleum odor.
BOILING POINT: 350-680 F (177-360 C)
FREEZING POINT: 0 F (-18 C)
VAPOR PRESSURE: 1 mmHg @ 20 C

VAPOR DENSITY: >1
SPECIFIC GRAVITY: 0.87-0.90
WATER SOLUBILITY: insoluble
VISCOSITY: 32.6-40.1 SSU @ 100 F

SECTION 10 STABILITY AND REACTIVITY

REACTIVITY:
Stable under normal temperatures and pressures in a closed container.

CONDITIONS TO AVOID:
Avoid contact with heat, sparks, flames, or other sources of ignition. Vapors may be explosive. Avoid overheating of containers; containers may violently rupture in heat of fire. Avoid contamination of water sources.

Trace amounts of hydrogen sulfide may be present in this product. There is a potential for accumulation of hydrogen sulfide in the head space of containers or in enclosed areas where the product is stored, handled or used.

INCOMPATIBILITIES:
DIESEL FUEL:
OXIDIZERS (STRONG): Fire and exposion hazard.

HAZARDOUS DECOMPOSITION:
Thermal decomposition products may include toxic oxides of sulfur and carbon.

POLYMERIZATION:
Hazardous polymerization has not been reported to occur under normal temperatures and pressures.

SECTION 11 TOXICOLOGY INFORMATION

DIESEL FUEL:
TOXICITY DATA: 7.5 gm/kg (marketplace sample) oral-rat LD50 (AETODY); >5 ml/kg (marketplace sample) skin-rabbit LD50 (AETODY).
CARCINOGEN STATUS: Human Inadequate Evidence, Animal Limited Evidence (IARC Group-2B). (See additional data)
LOCAL EFFECTS: Irritant- inhalation, skin.
ACUTE TOXICITY LEVEL: Slightly toxic by dermal absorption; relatively non-toxic by ingestion.
TARGET EFFECTS: Central nervous system depressant. Poisoning may also affect the liver and kidneys.
ADDITIONAL DATA: Animal studies have confirmed an association between the induction of cancer, primarily of the lung, and inhalation exposure to whole diesel exhaust. Limited epidemiologic evidence also suggests an association between occupational exposure to diesel engine emissions and lung cancer (NIOSH, 1988).

HEALTH EFFECTS
INHALATION:
DIESEL FUEL:
IRRITANT/NARCOTIC.

ACUTE EXPOSURE- Vapors or mist may cause respiratory tract irritation. A human exposure has resulted in immediate cough, dypsnea, cyanosis and unconsciousness for one hour. A productive cough with sputum smelling of diesel fuel persisited for 37 days. Chest X-rays showed diffuse shadowing, most prominent at the lung bases, which resolved slowly with treatment but was still present at day 37. High levels may also cause central nervous system excitation followed by depression with symptoms possibly including restlessness, confusion, ataxia, headache, dizziness, anorexia, nausea, vomiting, weakness, incoordination, stupor, delirium and coma.

CHRONIC EXPOSURE- Prolonged or repeated exposure may cause irritation. One individual exposed to diesel vapors in a truck cab developed nephrotoxic effects.

SKIN CONTACT:
DIESEL FUEL:
IRRITANT.

ACUTE EXPOSURE- May cause smarting, redness and irritation. A sample of diesel fuel applied to rabbits under a patch for 24 hours caused extreme irritation with severe erythema and edema with blistering and open sores.

CHRONIC EXPOSURE- Repeated or prolonged contact may cause defatting and drying of the skin resulting in irritation and dermatitis. Cutaneous hyperkeratosis has been described in engine drivers with occupational exposure to diesel fuel. Two individuals with topical exposure from washing hair or hands with diesel fuel developed acute renal failure; one also had gastrointestinal symptoms. Repeated applications to rabbit skin produced 67% mortality at 8 mL/kg. The primary causes of death were depression and anorexia which were induced by dermal irritation with infection, rather than systemic intoxication. Autopsy revealed effects on the liver and kidneys.

EYE CONTACT:
DIESEL FUEL:

ACUTE EXPOSURE- Liquid or vapor may cause slight irritation, although tests with one sample of diesel fuel in rabbit eyes was non-irritating.

CHRONIC EXPOSURE- Repeated or prolonged exposure may cause irritation.

INGESTION:
DIESEL FUEL:
NARCOTIC.

ACUTE EXPOSURE- May cause nausea, vomiting, cramping, diarrhea, and possibly symptoms of central nervous system depression. Aspiration of even small amounts during ingestion or vomiting may result in severe pulmonary irritation with coughing, gagging, dyspnea, substernal distress, and pneumonitis, pulmonary edema and hemorrhage, and death.

CHRONIC EXPOSURE- No data available.

SECTION 12 ECOLOGICAL INFORMATION

ENVIRONMENTAL IMPACT RATING (0-4): no data available

ACUTE AQUATIC TOXICITY: no data available

BIODEGRADABILITY: no data available

BIOCONCENTRATION FACTOR (BCF): no data available

LOG OCTANOL/WATER PARTITION COEFFICIENT: no data available

SECTION 13 DISPOSAL INFORMATION

Observe all federal, state and local regulations when disposing of this substance.

Disposal must be in accordance with standards applicable to generators of hazardous waste, 40 CFR 262. EPA Hazardous Waste Number D001.
100 pound CERCLA Section 103 Reportable Quantity.

SECTION 14 TRANSPORTATION INFORMATION

U.S. DEPARTMENT OF TRANSPORTATION SHIPPING NAME-ID NUMBER, 49 CFR 172.101:
Diesel fuel-NA 1993

U.S. DEPARTMENT OF TRANSPORTATION HAZARD CLASS OR DIVISION, 49 CFR 172.101:
3 - Flammable liquid

U.S. DEPARTMENT OF TRANSPORTATION PACKING GROUP, 49 CFR 172.101:
III

U.S. DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS, 49 CFR 172.101 AND SUBPART E:
None

U.S. DEPARTMENT OF TRANSPORTATION PACKAGING AUTHORIZATIONS:
EXCEPTIONS: 49 CFR 173.150
NON-BULK PACKAGING: 49 CFR 173.203
BULK PACKAGING: 49 CFR 173.241

U.S. DEPARTMENT OF TRANSPORTATION QUANTITY LIMITATIONS 49 CFR 172.101:
PASSENGER AIRCRAFT OR RAILCAR: 60 l
CARGO AIRCRAFT ONLY: 220 l

SECTION 15 REGULATORY INFORMATION

TSCA STATUS: Y

CERCLA SECTION 103 (40CFR302.4):	Y	100	pounds RQ
SARA SECTION 302 (40CFR355.30):	Y	500	pounds TPQ
SARA SECTION 304 (40CFR355.40):	Y	100	pounds RQ
SARA SECTION 313 (40CFR372.65):	N		
OSHA PROCESS SAFETY (29CFR1910.119):	Y	1500	pounds TQ
CALIFORNIA PROPOSITION 65:	N		

SARA HAZARD CATEGORIES, SARA SECTIONS 311/312 (40 CFR 370.21)
ACUTE HAZARD: Y

CHRONIC HAZARD: Y
RE HAZARD: Y
TIVITY HAZARD: N
EN RELEASE HAZARD: N

SECTION 16 OTHER

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DEERE & COMPANY
John Deere Road, Moline, IL 61265
1-800-822-8262

Material Data Safety Sheet

JOHN DEERE PRODUCT NAME: **GL5 Gear Lubricant (SAE 80W/90)**

DATA SHEET NO: 8503-40,040
LATEST REVISION DATE: 15 Feb. 1989
DEERE CODE: XJ
JDM PART NO: TY6252, TY6296
TY6382, TY6383

SECTION I - PRODUCT IDENTIFICATION

CHEMICAL NAME AND SYNONYMS: Lubricating Oil; Gear Oil
CHEMICAL FAMILY: Hydrocarbon FORMULA: Complex

SECTION II - HAZARDOUS INGREDIENTS

INGREDIENT	PERCENT	TLV/PEL	V.P.	CAS.#
Solvent refined, hydro-treated residual oil	40-90	5 mg/m^3*	-	64742570
Solvent refined, hydro-treated dewaxed heavy paraffinic dist.	5-30	-	-	64742650
Severly hydrotreated heavy naphthenic distillate	10-40	-	-	64742525
Polymeric additive in oil (poly-methacrylate)	10	None	-	None

*for product

SECTION III - PHYSICAL DATA

BOILING POINT: > 700°F
% VOLATILE VOLUME: N.A.
VAPOR DENSITY: N.A.
APPEARANCE/ODOR: Amber/petroleum

SP. GRAVITY (WATER=1): 0.90
EVAPORATION RATE: N.A.
SOLUBILITY IN WATER: Insoluble
N.A. - not available

SECTION IV - FIRE & EXPLOSION HAZARD DATA

FLASH POINT: 360° F
FLAMMABLE LIMIT - LEL: N/A
EXTINGUISHING MEDIA: Water fog, foam dry chemical, carbon dioxide, or halogenated agents.
SPECIAL FIRE FIGHTING PROCEDURES: Do not use a direct stream of water. Product will float and can be reignited on surface of water. Cool fire exposed containers with water. Use NIOSH approved self-contained breathing apparatus.

UNUSUAL FIRE & EXPLOSION HAZARDS: None

Recycled Paper

SECTION V - HEALTH HAZARD DATA

EXPOSURE LIMIT: See Section II - Hazardous Ingredients
EFFECTS OF OVEREXPOSURE: Exposure to vapors or mists of this product may cause mild upper respiratory tract irritation. Prolonged or repeated contact may cause various skin disorders such as dermatitis, oil acne, or folliculitis. Eye contact is minimally irritating. Effects of ingestion are expected to be relatively non-toxic. Preexisting skin & respiratory disorders may be aggravated by exposure to this product.
EMERGENCY & FIRST AID: Eyes - flush with water 15 minutes. Skin - remove contaminated clothing; wash skin with soap and water; if material is injected under the skin, do not wait for symptoms to develop - get medical attention promptly to prevent serious damage. Inhalation - remove victim to fresh air and provide oxygen if breathing is difficult. Ingestion - do NOT induce vomiting. In all cases seek medical attention.

SECTION VI - REACTIVITY DATA

STABILITY: Stable
INCOMPATIBILITY: Avoid open flame, and oxidizing materials
HAZARDOUS POLYMERIZATION: Will not occur
DECOMPOSITION PRODUCTS: Dependent on combustion conditions. A complex mixture of airborne solid liquid and gas will evolve when this material undergoes pyrolysis or combustion. Oxides of carbon, sulfur, phosphorous, and other unidentified organic compounds may be formed.

SECTION VII - SPILL OR LEAK PROCEDURE

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED: Dike and contain. Use vacuum or an absorbent such as clay or sand to pick up. Flush area with water to remove trace residue. NOTE: This product is classified as an oil under the Clean Water Act. Spills, entering surface waters or any watercourse or sewer leading to surface waters, must be reported to the National Response Center 800-424-9802.
WASTE DISPOSAL METHOD: In accord with federal, state, and local regulations

SECTION VIII - PROTECTIVE EQUIPMENT INFORMATION

VENTILATION: Local exhaust to keep TLV/PEL below acceptable levels
RESPIRATOR: NIOSH approved as needed
EYE WEAR: Recommended
GLOVES: Recommended to minimize skin contact
OTHER:

SECTION IX - SPECIAL PRECAUTIONS

Minimize skin contact. Wash with soap and water before eating, smoking, or using toilet facilities. Launder contaminated clothing before reuse. Properly dispose of contaminated articles including shoes that cannot be cleaned. Store in a cool, dry place with adequate ventilation. Keep away from open flames. Keep away from children.

SECTION X - DATA PREPARATION

NAME: T. M. Snyder, CIH
TITLE: Industrial Hygienist
SIGNATURE: T. M. Snyder
DATE: January 26, 1998

The information contained herein is believed to be accurate. However, no warranty is expressed or implied regarding the accuracy of these data or the results to be obtained from the use thereof. Vendor assumes no responsibility for injury to vendee or third persons proximately caused by the material if reasonable safety procedures are not adhered to as stipulated in the data sheet. Furthermore, vendee assumes the risk in use of the material.

ATTACHMENT B
Daily Safety Log

TOLTEST, INC.

NSWC Crane, Indiana

"Plan of the Day/Safety Brief"

Associate	Signature

Activities	*Activities*	*Activities*

"Daily Hazard Communication Brief"

ATTACHMENT C
Equipment Inspection Log

EQUIPMENT INSPECTION FORM

OPERATORS DAILY PRE-OP, MAINTENANCE & SAFETY INSPECTION

Type:	Hours:		Miles:	- - 00	Op:
Inspection Item	**OK**	**N/A**	**Adjustments/Repairs Made or Required**		
Service, Parking & Emergency Brakes					
Steering Mechanism					
Tires, Wheels, Nuts					
Lights, Reflectors					
Coupling Devices					
Operating Controls					
Windshield Wipers					
Horn & Backup Alarm					
Seat Belts/Shoulder Harness					
Fire Extinguisher					
Mirrors					
Hydraulic System Operation					
Fluid Levels (Oil & Water)					
Tracks					

ATTACHMENT D

Incident Forms

Tracking #_____

Accident & Incident Report

___ Personal Injury/Illness
- __ First Aid
- __ Other: _____

___ Safety/Non-Injury Incident
- __ Near Miss
- __ Vandalism / Property Damage
  - __ Personal Property
  - __ Company Property

___ Other: _____

___ Vehicle Incident; Vehicle #:_____
- __ Accident
- __ Body Damage
- __ Theft

___ Equipment Damage
- __ Lost
- __ Stolen

Associates Name: _____ SS#: _____

Date Incident Occurred: _____ Time of Incident: ____ A.M. P.M.

Supervisor's Name: _____ Date and Time Reported: _____

Project Manager's Name: _____

Date of Hire: _____ Time in Present Occupation: ____ years ____months

Names of Witnesses to the Incident: _____

_____

In case of accident, were the police called: ___ Yes ___ No If yes, who responded:

_____

Describe the Events leading to the Incident: _____

_____

Task at which the associate was engaged (Describe any tools or equipment being used):

_____

Description of the work area: _____

_____

Description of the incident: _____

_____

_____

Did the incident take place on the premises: ___ Yes ___ No Location: _____

_____

Tracking #_____

Did associate receive medical care? ___ Yes ___ No Number of days NOT worked? _____

If yes, where (Place, city, and phone #): _____

_____

Name of the attending physician: _____

Type of injury: _____ Part of body injured: _____
(Sprain, Strain, Cut, Abrasion, Contusion, Break, etc.)

First aid and/or medical treatment provided (be specific): _____

Have you ever injured this area before? ___ Yes ___ No If yes, when: _____

Safety Officer's Comments: _____

_____

_____

_____

Corrective action (Preventative, Avoidance, Engineered changes): _____

_____

_____

_____

Signature of Associate: _____ Date: _____

Signature of Departmental Safety Officer: _____ Date: _____

Signature of Corporate Safety Officer: _____ Date: _____

HUMAN RESOURCES DEPARTMENT

ABRA Case #: _____ Lost Time: Y N Restrictions: _____

Recordable: Y N # Days Away: ____ # Days Restricted: _____

Out of State: Y N Filed with BWC: Y N Date filed: _____

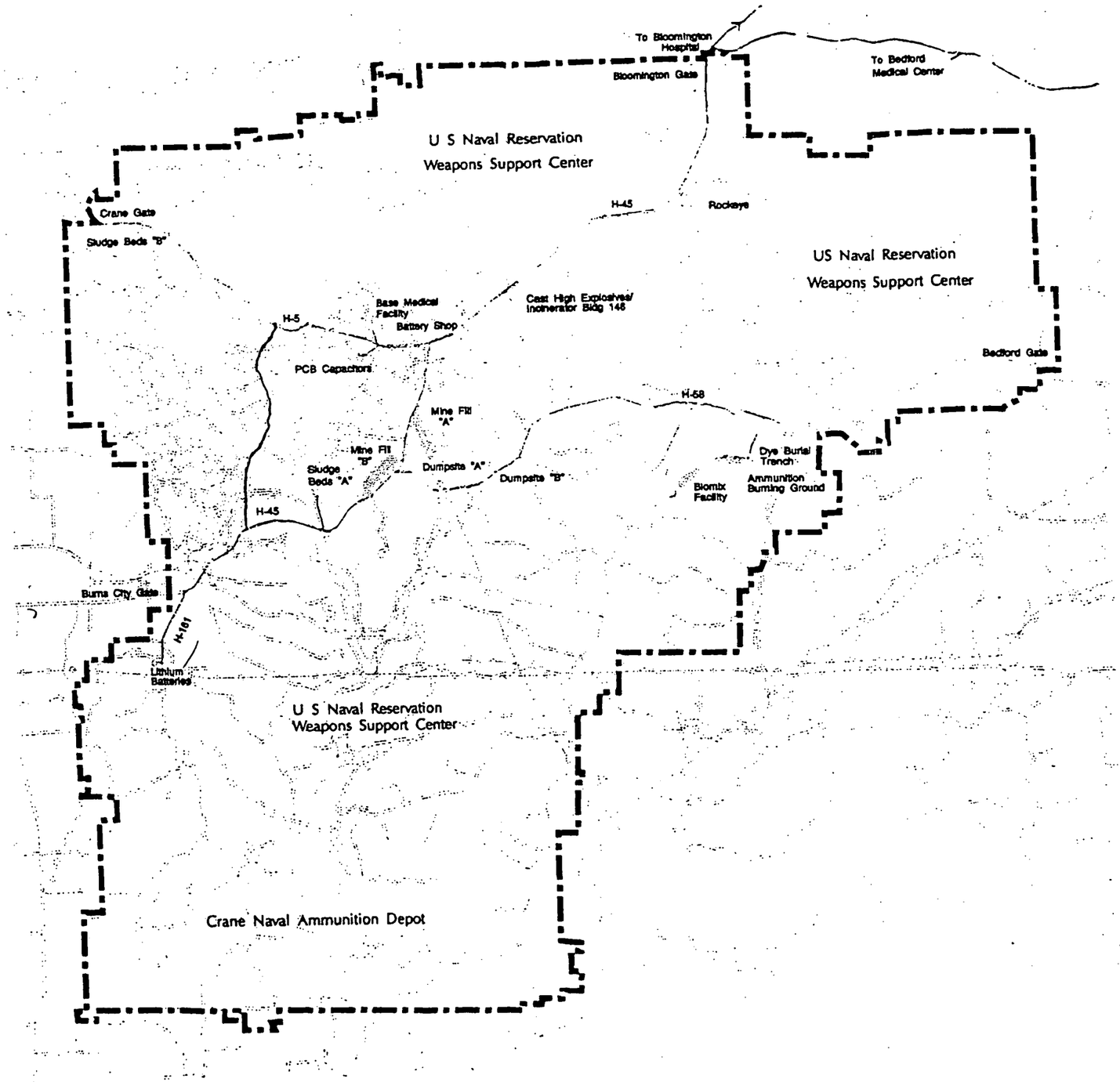
BWC Claim #: _____ Date Closed: _____

Original: ___ Health and Safety File

CC: ___ Associate ___ Dept. Safety Rep. ___ Worker's Comp File

ATTACHMENT E

Directions and Area Maps to Hospitals



Directions to the Nearest Medical Facility

Legend

Primary Route

Naval Reservation Boundary

Directions to NSWC Medical Department on site:

The NSWC Medical Department manages and coordinates the on site ambulance service. Located in Building 12 off of H-2 just north of H-5.

Directions to Bedford Medical Center:

From Main Gate, head east on Highway 58 to the city of Bedford, left onto 16th street. Distance to hospital is approximately 20 miles.

Directions to Bloomington Hospital:

Exit Base on H5-45 through the Bloomington Gate, follow Highway 45 North to Bloomington. At Highway 45 and Highway 37, continue going straight over the bypass (Bloomfield Road), follow Bloomfield road north which turns into 2nd Street. Follow 2nd Street and hospital is on your right.

APPENDIX B

SAMPLING AND ANALYSIS PLAN

FINAL

SAMPLING AND ANALYSIS PLAN FOR SWMU 12/14

MINE FILL A BATTERY SITE CLEANUP AT THE NAVAL SURFACE WARFARE CENTER, CRANE, INDIANA

CONTRACT NO. N68950-96-D-0052
DELIVERY ORDER # FC04
TOLTEST PROJECT NO. 37826.01

FEBRUARY 2001

Submitted to:

OFFICER IN CHARGE OF NAVFAC CONTRACTS
NAVAL SURFACE WARFARE CENTER
BUILDING 2516
300 HIGHWAY 361
CRANE, INDIANA 47522-5082

Submitted by:

TOLTEST, INC.
1915 NORTH 12TH STREET
P.O. BOX 2186
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(419) 241-7175

TABLE OF CONTENTS

Page No.

1.0 PROJECT DESCRIPTION B-1
- 1.1 Introduction B-1
- 1.2 Site Description B-1
- 1.3 Site Physical Characteristics B-1
- 1.4 Project Sampling Objectives B-1
- 1.5 Data Quality Objectives B-2

2.0 PREPARATION FOR SAMPLING B-3
- 2.1 Training Requirements B-3
- 2.2 Pre-Sampling Tasks B-4
- 2.3 Sampling Locations B-4
- 2.4 Analytical Requirements B-5
  - 2.4.1 Soil Area B-5
  - 2.4.2 Battery Area B-6
  - 2.4.3 Background B-7
- 2.5 Project Target Parameters and Intended Data Usage B-7

3.0 SAMPLING PROCEDURES B-8
- 3.1 Sampling Procedures B-8
- 3.2 QC Sample Procedures B-8
- 3.3 Sample Disposal B-8
- 3.4 Sample Identification System B-9
- 3.5 Field Activity Documentation/Logbook B-10
- 3.6 Sample Containers, Sample Preservation, and Maximum HoldingTime B-10
- 3.7 Decontamination Procedures B-11

4.0 CUSTODY PROCEDURES B-12
- 4.1 Field Custody Procedures B-12
- 4.2 Sample Packaging and Shipment Procedures B-13

REFERENCES B-14

TABLE OF CONTENTS (Continued)

TABLES

Table 1.0 Battery Area Constituent List B-6
Table 2.0 Sample Container Prevention, and holding time requirements B-10

FIGURES

Figure 1 Pre-excavation Sample Location Map

ATTACHMENTS

Attachment A Michigan Dept. of Environmental Quality Verification of Soil Remediation Guidance Document
Attachment B Appendix IX Constituent List
Attachment C Battery Material Safety Data Sheets
Attachment D Soil Screening Levels
Attachment E Soil Sampling Standard Operating Procedure

1.0 PROJECT DESCRIPTION

1.1 Introduction

TOLTEST, Inc. (TOLTEST) has prepared this Sampling and Analysis Plan (SAP) for interim measures cleanup defined in the scope of work letter dated April 13, 1999, under Contract #N68950-96-D-0052 and Delivery Order FC04. This SAP describes the field activities that must be performed and defines the procedures and methods that must be used to collect field measurements and samples. It also discusses quality assurance/quality control (QA/QC) samples, and requirements for sample chain of custody, documentation, and shipping.

This project includes furnishing all labor, transportation, supervision, material and equipment, and performing all operations in connection with the excavation, sampling, and disposal of the battery disposal area and a potentially contaminated soil area at SWMU 12/14, Mine Fill A Battery Site Cleanup. Since this is an interim measure, the scope of work for this contract does not address the potential for groundwater contamination. This scope addresses only the removal of the potential source of contamination.

1.2 Site Description

A detailed discussion of the site description is presented in Section 1.1 of the Interim Measures Work Plan.

1.3 Site Physical Characteristics

A detailed discussion of the site physical characteristics is presented in Section 1.2 of the Interim Measures Work Plan.

1.4 Project Sampling Objectives

The sampling objectives are to collect pre-excavation samples for site characterization, characterization of contaminated soil for disposal purposes, and collect post-excavation confirmatory samples. The groundwater sampling is not addressed in this scope of work.

1.5 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements specifying the quality of the data required to support decisions made during interim measures cleanup, and are based on the end uses of the data to be collected. This project will utilize NEESA Level C for all off-site analytical work.

The primary DQOs for sampling described in this SAP are to produce data of sufficient quality and quantity in order to determine if excavated areas meet cleanup goals (discussed in section 2.5 of this plan) and to characterize the excavated soil for the proper disposal. The data collected for soil disposal must be of acceptable quantity and quality to demonstrate compliance with established Resource Conservation and Recovery Act (RCRA) requirements and/or specific disposal facility requirements. These objectives will be achieved by controlling sample collection, sample custody, sample analysis, sample data review, and data reporting. Sample collection procedures are described in Section 3 of this plan.

Sampling and analytical methods will be consistent with standard methods outlined by the United States Environmental Protection Agency (USEPA) and meet CLP Level IV criteria. Laboratory data will be calculated and reported in a manner consistent with USEPA approved analytical procedures and with other TOLTEST programs that report similar data. Data acquisition will be amenable to evaluation of precision, accuracy, representatives, comparability, and completeness as discussed in Section 4 of the Quality Assurance Project Plan (Appendix C of the Battery Site Work Plan).

Remedial goals, methodologies, and laboratory reporting limits for all analyses are discussed in Section 8.0 of the QAPP (App. C to the WP).

If the laboratory is unable to meet these reporting goals, TolTest will contact the Laboratory Project Manager to discuss corrective action measures. If after implementation of corrective action measures the laboratory is still unable to meet project reporting goals, TolTest will contact the ECOTR to discuss resolution options.

2.0 PREPARATION FOR SAMPLING

This section includes procedures and steps to be followed prior to sampling and includes training requirements, pre-sampling tasks, sampling locations, analytical requirements, and project target parameters and intended data usage.

2.1 Training Requirements

Personnel responsible for collecting field samples will be required to read this SAP and will be familiar with all required methods and procedures. The field sampling crew and the Quality Control personnel performing inspections will be trained prior to commencing field activities. Training will be verified as part of TOLTEST'S review process. Personnel in charge of reducing, evaluating, and reviewing the laboratory field data must have a working knowledge of the analytical methods employed.

All field personnel will be trained as described in the SSHP (Appendix A of the Battery Site Work Plan). Required training includes the following:

- Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste and Emergency Response Training per 29 Code of Federal Regulations (CFR) 1910.120, updated by annual 8-hour refresher training.
- OSHA 8-hour supervisor's training course in Hazardous Waste Site Safety per 29 CFR 1910.120(e) (field management personnel only).
- Site specific training.
- Respiratory training.
- Two on-site personnel will be required to have First Aid and CPR training.
- The individual responsible for the safe package, labeling and shipping of hazardous environmental samples must meet the training requirements specified in United States Department of Transportation (USDOT) 49 CFR 172.700, Subpart H.

2.2 Pre-Sampling Tasks

Prior to sample collection, the following tasks will be completed:

- Review information, such as the Work Plan, SSHP, QAPP, previous site characterization reports, disposal records, etc., to gain general knowledge of the potential materials to be encountered.
- Ensure appropriate staging areas have been properly constructed. Sufficient room should be allowed for exclusion zones, contamination reduction zones, and support zones around staging areas. Verify that excavation permits have been obtained.
- Carefully review the SSHP and Work Plan. All field personnel will attend a health and safety meeting at the beginning of each day and prior to starting a new task. Field personnel will be asked to document that training was completed, and that they will abide by the provisions stated in Appendix A of the Battery Site Work Plan.
- Inventory all supplies. Maintain careful records of calibration data for all monitoring equipment. Check supply list daily to determine if additional items are needed. A general list of sampling and other field equipment is provided in Table 2.0 of the Work Plan.
- Obtain all necessary documentation materials, including, field logbooks, sample labels, chain of custody forms, etc.
- Collect the applicable health and safety equipment as defined in Appendix A of the Battery Site Work Plan.
- Confirm that utilities have been marked prior to sampling activities.
- Confirm that unexploded ordnance clearance has been received.

2.3 Sampling Locations

To determine the number of sample points relative to the size of the area to be sampled, the Verification of Soil Remediation Guidance Document[1] (Guidance Document) was referenced. The Guidance Document is provided as Attachment A to this plan. The size of the soil area is approximately 2355 square feet consisting of several soil mounds, none exceeding four feet in height. The size of the battery area is approximately 3500 square feet. Following the Guidance Document guidelines, a total of 11 pre-excavation grab samples will be obtained from the site: five from the soil area and six from the battery area.

The approximate location of the soil samples is depicted in Figure 1, Pre-Excavation Sample Location Map. The location of sample points will be biased to areas of suspected contamination as discussed in the Guidance Document. The samples from the soil mounds will be obtained at varying depths from the surface to deep within the mounds. Samples from the battery area will be obtained from the surface to a depth of 12 inches. All samples will be grab samples, no composite samples will be obtained.

Pre-excavation sample points will be identified in the field with pin flags with the sample identifier written on the flag in indelible ink.

Once the identified areas of contamination have been excavated, confirmatory (post-excavation) sampling activities will proceed. The area of excavation will be measured and the number of floor samples to be obtained will be determined by the size of the excavation using the Guidance Document as a reference. Sidewall samples will be obtained if the excavation is one foot deep or deeper. The number of sidewall samples to be obtained will be based on the total square footage of the sidewalls. No less than one sample will be collected per sidewall. Post-excavation samples will be collected from the surface to 6 inches deep.

Post-excavation boundaries and sample points will be surveyed and mapped at the conclusion of the project.

2.4 Analytical Requirements

2.4.1 Soil Area

One sample from the soil area will be analyzed for explosives by EPA Method 8330 and Appendix IX[2] constituents consisting of metals, cyanide, sulfide, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), herbicides, and pesticides/PCBs. No analysis for dioxins/furans will be completed. The Appendix IX list can be found as Attachment B to this plan. Analysis for VOCs will conform to SW-846, method 8260/5035 for EnCore samplers. The remaining four samples from the soil area will be analyzed for Mine Fill A constituents (based on the Biofacility QAPP requirements) of explosives, metals, and VOCs. These constituents were chosen based on the

reasonable assumption that the soil in this area originated from Mine Fill A and the fact that, according to Crane EPD, there is no history of dioxin/furan contamination at Crane.

Soil that is shown to be contaminated with Mine Fill A constituents only will be handled and processed according to requirements set forth in the Biofacility Work Plan and QAPP.

2.4.2 Battery Area

Pre-excavation samples from the battery area will be analyzed for toxic characteristics and metals as listed in Table 1.0.

Table 1.0
Battery Area Constituent List

CONSTITUENT	METHOD
Ignitability	1010
Corrosivity	EPA 150.1
Reactivity	Cpt. 7, SW 846
RCRA Metals	6010 & 7471
Cobalt	6010
Lithium	6010
Manganese	6010
Nickel	6010
Zinc	6010

The additional metals chosen (over and above the 8 RCRA metals) were based on constituents listed in several Material Data Safety Sheets (MSDSs) for batteries, and Appendix IX metals. Copies of the MSDSs are provided as Attachment C to this plan.

Analysis for disposal characterization (to include TCLP) will be completed if total metals analysis indicates that the contamination, if present, exceeds established cleanup levels.

Post-excavation samples will be analyzed for total metals only.

2.4.3 Background

Analysis of background samples is discussed in Section 2.5 below.

2.5 Project Target Parameters and Intended Data Usage

Intended data usages are to achieve closure of the excavation pending removal of impacted soils above cleanup goals, and characterization of excavated soil for disposal. The analytical results from pre-excavation samples will be compared with the Risk Based Soil Screening Levels[3] (SSLs), the EPA Region9 or Region 5 Preliminary Remediation Goals (PRGs), and Ecological Data Quality Levels (DQLs). The SSLs and Region 9 PRGs are provided as Attachment D to this plan. Analytical requirements are discussed further in Section 8.0 of the QAPP. Inorganic constituents will also be compared with the background levels determined from the Basewide Background Soils Study. Background levels of organic compounds are assumed to be zero.

All analytical data will be presented to the NSWC Crane Environmental Contracting Officer Technical Representative (ECOTR) who will determine the appropriate cleanup goals for the site. Cleanup will be attained when post-excavation confirmatory sample results are shown to be below the established cleanup goals.

3.0 SAMPLING PROCEDURES

The sampling procedures to be used will be consistent with the purpose of this project. This section outlines sampling procedure information. Detailed procedures for obtaining soil samples are included as Attachment E, Standard Operating Procedures 1.0: Soil Sampling.

3.1 Sampling Procedures

All samples will be collected by the Environmental Specialist or Environmental Technician. The samples from the mounds in the soil area will be obtained at varying depths from the surface to deep within the mounds, however the total depth of each sample will be 12 inches. Samples from the battery area will be obtained from the surface to a depth of 12 inches. All samples will be grab samples, no composite samples will be obtained.

All samples will be labeled and tagged with the sampler's name, time and date of collection, sample number, and analytical parameter. All samples collected in the field will be placed directly on ice in a cooler to await shipping to the off-site laboratory.

When all the known contaminated materials have been removed from the excavation, confirmation samples will be collected.

3.2 QC Sample Procedures

Quality Control samples will consist of field duplicates, matrix spike/matrix spike duplicates (MS/MSD), rinse blanks, and trip blanks. One field duplicate will be collected for every twenty or fewer regular samples. One sample for MS/MSD analysis will be collected for every 20 samples collected ;however at least one sample per shipment will be identified for MS/MSD purposes. Significant changes in the location or number of samples will be approved by the ECOTR.

3.3 Sample Disposal

Samples will be retained at the laboratory under control of the sample custodian for 30

days after analysis. Upon approval by the TOLTEST PM, the samples will be disposed following applicable state and federal regulations.

3.4 Sample Identification System

The sample identification system consists of site-specific sample numbers. The first and second set of three characters represents the excavation site; the third set of three characters represents the sample type; the next three numbers represent the unique sample number; the last two characters will be used to identify field duplicates.

Excavation site
MFA – Mine Fill A
BAT – Battery Site

Sample Type
PES – Pre-Excavation Soil
CFS – Confirmatory Soil

Sample Number
- Unique Sample Number

An example of a sample identifier would be as follows: MFA-BAT-PES-001-FD, which is Mine Fill A, Battery Dump, Pre-excavation sample number 001, field duplicate.

QC Sample Identifiers

The first and second set of three characters represents the excavation site; the next two characters represent the QC designation (TB = trip blank, RB = rinse blank); and the last six numbers represent the month, day, and year (ex. 070399 = July 3, 1999). An example of a sample identifier would be as follows: MFA-BAT-RB070399. All sample locations and identifiers will be recorded and documented in field logs and sample location maps.

3.5 Field Activity Documentation/Logbook

All information pertinent to the environmental samples, including specific field collection data and laboratory observations will be recorded in permanently bound notebooks. TOLTEST'S contract laboratory also employs a specific information management system to assist in tracking the progress of each sample through the analytical process.

3.6 Sample Containers, Sample Preservation, and Maximum Holding Time

All samples collected will be preserved according to USEPA and/or Contracting Officer protocols established for the parameters of interest. Appropriate measures will be taken to ensure that storage requirements with respect to temperature are maintained during transport to the laboratory and prior to log-in and storage at the laboratory. Table 2.0 summarizes sample containers, preservation, and holding time requirements. Samples will be analyzed in the laboratory on a standard turn-around-time (TAT) of 21 days unless expedited TAT is requested.

Table 2.0

Sample Container, Preservation, and Holding Time Requirements

Matrix	Analysis	Container	Preservation	Holding Time
Soil	Metals; SVOCs, pesticides/PCBs; Sulfide, Cyanide, and Explosives	Two 8 oz. Wide-mouth glass jar, per sample aliquot	Cool to 4° C	6 months for metals, 14 days for all other analyses
Soil	VOCs	3 EnCore Samplers	Cool to 4° C	48 hours

3.7 Decontamination Procedures

All sample containers will be certified clean, EPA-approved, laboratory-prepared glass sample jars with Teflon®-lined lids. Any sampling equipment, which is not disposable, will be decontaminated according to the following procedures;

- Wash in soapy water (alconox or equivalent)
- Rinse in potable water
- Rinse with 10% nitric acid
- Rinse with reagent grade water
- Air dry

The amount of decontamination water generated will be kept to less than 2 gallons per sampling event. Waste decontamination water will be brought to the TOLTEST on-site facility and stored in a 500-gallon poly tank along with decontamination water generated from other TOLTEST projects at NSWC Crane. When full, the contents of the poly tank will be sampled and characterized for disposal purposes.

4.0 CUSTODY PROCEDURES

Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence file. Laboratory analysis and final evidence files are discussed in detail in sections 6.1 and 6.2 of Appendix C to the Battery Site Work Plan.

4.1 Field Custody Procedures

Field logbooks will provide the means of recording sampling activities. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory. Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel but will be stored in the document control center when not in use. The project-specific document number will identify each logbook.

The title page of each logbook will contain the following:

- Person to whom the book is assigned,
- Logbook number,
- Project name,
- Project start date, and
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the dates, start time, weather, names of all project team members present, and level of personal protection being used will be entered. The names of visitors to the site and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded in the logbook. All entries will be made in ink, signed, and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark signed and dated by the sampler. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station will be recorded. All equipment used to make measurements will be identified, along with the date of calibration. The equipment used

to collect samples will be noted along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. The number of the photographs taken of the station, if any, will also be noted.

4.2 Sample Packaging and Shipment Procedures

The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain of custody intact.

a. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

b. All bottles will be identified by use of sample labels with sample numbers, sampling locations, date/time of collection, sampler's initials and type of analysis.

c. A properly completed chain of custody form will accompany samples. The sample numbers and locations will be listed on the chain of custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of sample from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

d. Samples will be properly packaged on ice at 4° C for shipment and dispatched to the appropriate laboratory for analysis with a separate signed custody record enclosed in and secured to the inside top of each sample box or cooler. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is sealed with strapping tape in at least two locations.

e. The chain of custody record identifying the contents will accompany all shipments. The original record and yellow copies will accompany the shipment and the pink copies will be retained by the sampler for returning to the project file.

f. Samples will be transported by overnight carrier to the laboratory the same day the samples are collected in the field.

REFERENCES

1. Verification of Soil Remediation Guidance Document, Part 1, State of Michigan, Department of Environmental Quality, April 1994, Revision 1.

2. Appendix IX, 40 Code of Federal Regulations, Part 264, Subpart F, 1987.

3. Soil Screening Guidance: Technical Background Document (OSWER Publication 9355.4-17A; EPA/540/R-95/128; May 1996)

FIGURES

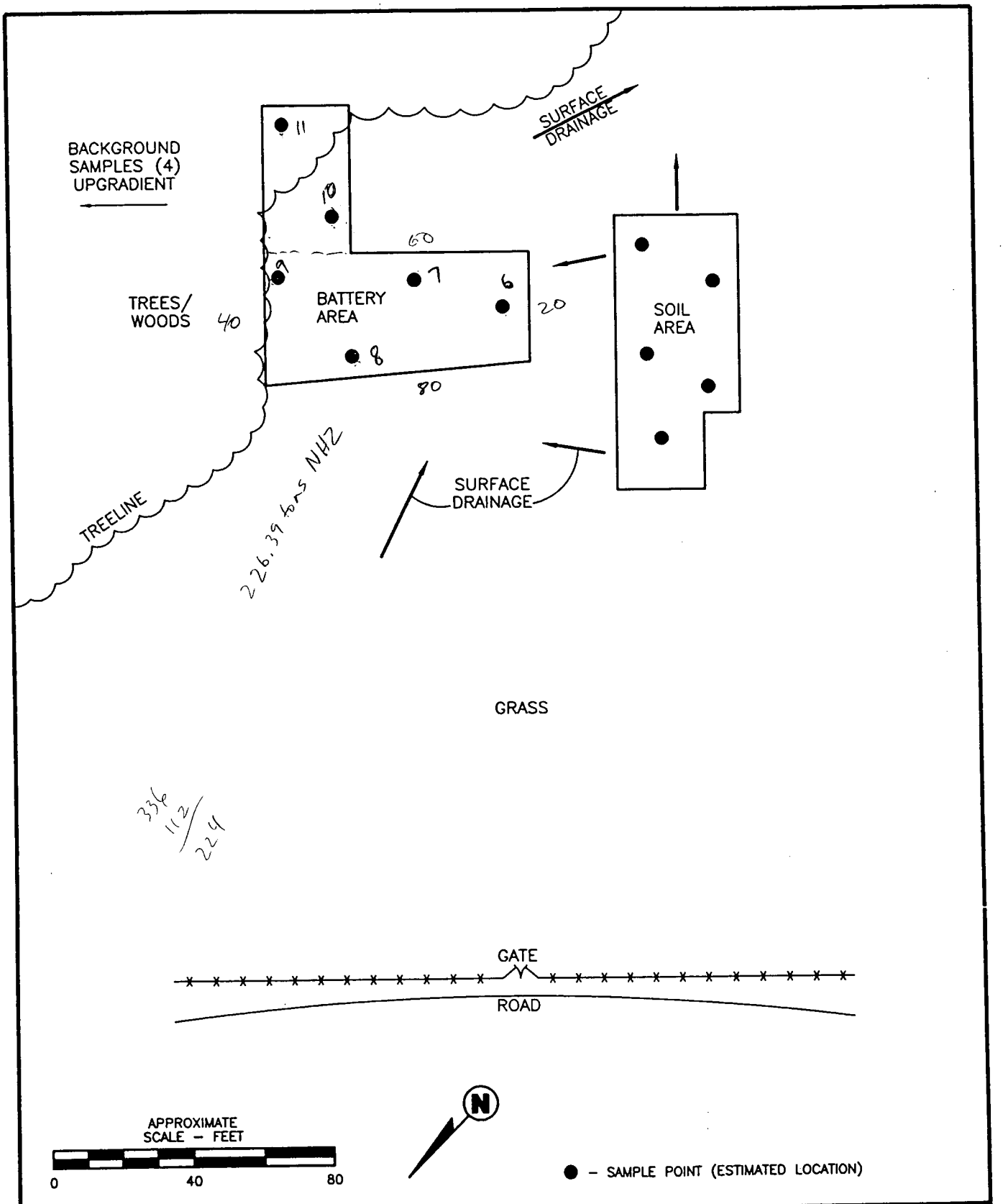

FIGURE 1 PRE-EXCAVATION SAMPLE LOCATION MAP BATTERY SITE CLEANUP NAVAL SURFACE WARFARE CENTER, MINE FILL A CRANE, INDIANA	DRAWN MRC\9-21-99	CHECKED
	REVISED	APPROVED
	JOB NO. 37826.01	TOLTEST, INC.
	DRAWING NUMBER 378261	

ATTACHMENT A

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY VERIFICATION OF SOIL REMEDIATION GUIDANCE DOCUMENT

ENVIRONMENTAL RESPONSE DIVISION

PLEASE NOTE: THE MATHEMATICAL FORMULAS IN THIS DOCUMENT ARE NOT DISPLAYED CORRECTLY IN HTML. IF YOU NEED TO USE THESE FORMULAS, PLEASE REQUEST A PAPER COPY OF THIS DOCUMENT.

GUIDANCE DOCUMENT

VERIFICATION OF SOIL REMEDIATION

ENVIRONMENTAL RESPONSE DIVISION

WASTE MANAGEMENT DIVISION

APRIL 1994, Revision 1

MICHIGAN DEPARTMENT OF NATURAL RESOURCES

GUIDANCE DOCUMENT FOR VERIFICATION OF SOIL REMEDIATION

EXECUTIVE SUMMARY

The document provides guidance for sampling soils to verify that soil contamination has been remediated to Type A or Type B criteria in accordance with Act 307 P.A. 1982, as amended. This document is not designed to either guide investigations to determine whether a release has occurred or the nature and extent of an identified release, nor to guide due diligence by a potential property owner. Issuance of this guidance document does not invalidate remedial action plans (RAPS) or clean-ups previously conducted and approved by the DNR.

Soil sampling and analyses to verify that site remediation is complete can result in two basic errors.

Declaring a site clean when it is contaminated

Declaring a site contaminated when it is clean

A soil sampling plan submitted to the DNR must minimize these errors. The guidance document presents acceptable methods for verifying soil remediation. It contains guidance on soil sampling protocols and documentation necessary to characterize and verify cleanup of contaminated soils. The document provides recommended procedures for establishing soil background concentrations, sampling grids, chemical constituent evaluations, statistical comparisons, verifying excavation and in-situ and ex-situ remedies, evaluating treated soils, and soil characterization. The recommended procedures are **not *absolutes***. Other methods are available to verify soil remediation. The Department of Natural Resources will evaluate other sampling and statistical strategies on a case-by-case basis.

The guidance document is divided into two parts:

Part 1 contains guidance for small site cleanup verification (less than 10,890 square feet--<.25 acre). It is a "biased" sampling strategy recommending soil sampling from areas most likely to contain

contamination.

Part 2 contains guidance for soil characterization and cleanup verification of medium and large sites (greater than 10,890 square feet-->.25 acre). It is a statistical random sampling strategy that minimizes biases in sampling.

Both sampling strategies require discrete soil samples. Compositing samples for cleanup verification is not accepted without prior DNR approval.

The guidance document contains verification checklists and reporting sections. The reporting sections should be carefully followed in reporting sampling rationale.

Reader's Note: Questions regarding this guidance document should be directed to Department staff you are currently working with for your project or site.

APRIL 94, Revision 1

TABLE OF CONTENTS--DNR GUIDANCE DOCUMENT VERIFICATION OF SOIL REMEDIATION

PART 1--Small Site Soil Cleanup Verification, Introduction 1

Verifying Excavation Remedies 2-4

Sample Locations 2

Number of Samples 3-4

Number of Excavation Floor Samples 3

Number of Excavation Sidewall Samples 3-4

Verifying In-Situ and Ex-Situ Soil Remedies 4

Sample Analysis 4

Background Soil Samples 5-8

Establishing Soil Background 5

Statistical Analysis for Establishing Background Concentrations 6-8

1. *Mean Plus 3 Standard Deviation Approach* 6-7

2. *Other Statistical Procedures for Establishing Background* 8

Procedures for Non-Detect Values 8

Report for Small Site Verification 9

PART 2--Medium and Large Site Soil Cleanup Verification, Introduction 10

Establishing Grid Interval 11-14

1. *Basic Strategies* 11-12

2. *Setting the Grid* 12

3. *Variations on Basic Strategy* 13-14

Sampling of Grid 14

Establishing Soil Background 14-15

Statistical Analysis for Establishing Background Concentrations 15-18

1. *Mean Plus 3 Standard Deviation Approach* 15-17

2. *Tolerance Limit* 17

3. *t-tests* 18

4. *Other Statistical Procedures for Establishing Background* 18

Procedures for Non-Detect Values 18

Statistical Evaluation of Data 18

Grid Approach to Additional Remediation 19

Two-Dimensional Node Sampling Excavation Grid 19

Two-Dimensional Subcell Sampling Excavation Grid 19

Three-Dimensional Cleanup Verification 19

Batch Sampling for Ex-Situ Treatment Processes 19

Disposal Options 20

Report for Medium & Large Site Verification 20

RCRA Clean Closure Certification Checklist 20

Cited References 42

ATTACHMENTS--DNR GUIDANCE DOCUMENT VERIFICATION OF SOIL REMEDIATION

1 Guide to Sample Bias 21-22

2 Sampling Protocol for Characterizing

Waste/Treatment Levels 23-38

Strategies for Evaluating Treated

Soils and Waste Materials 23

Statistical Sampling Strategies 24

Random Numbers Table 25-27

Sampling Grids 28

Statistical Evaluation of Waste/Treatment Characterization Samplings ...29-31

Example: Calculation of Confidence Limits & Lamda Calculation 32-34

Table: Cumulative t Distribution 35

Table: Number of Observations for t test of Mean 36

Sampling Process Streams 37

Random Time Waste Sampling Example 38

3 Tolerance Factors (K): [NTIS Document PB89151047, Table 5] 39

4 Waste Management Division's Clean Closure Certification Checklist 40-41

DNR--GUIDANCE DOCUMENT, PART 1

- 1 - SMALL SITE SOIL CLEANUP VERIFICATION (LESS THAN 10,890 SQUARE FEET)

Part 1 of this document is a guide for a biased sampling strategy to verify that soil contamination has been remediated at sites no greater than 0.25 acres (small sites). Soil sampling and analyses to verify that a site remediation is complete can result in two basic errors.

- Declaring a site clean when it is contaminated
- Declaring a site contaminated when it is clean

A soil sampling plan submitted to the DNR must minimize these errors. Part 1 presents a biased sampling method of verifying soil remediation at small sites. The biased sampling approach specified in this guidance recommends soil sampling from areas most likely to still exceed cleanup criteria. The location of the soil sample points relies on a site specific analysis of the released or contaminant distribution and the soil types encountered. The remediation is verified using a point by point comparison of sample values with the appropriate cleanup criteria. If the cleanup criteria are exceeded at any sampled point, the biased sampling methodology may require additional remediation at that point until the criteria are met. Verification of cleanup utilizing the biased approach should generally require fewer samples to demonstrate attainment than by using the unbiased approach. DNR will evaluate other sampling and statistical strategies on a case-by-case basis.

Any biased sampling plan, whether presented in the guidance document or some other geostatistical approach, requires professional judgment. Therefore, documentation and the rationale used to select sample locations are extremely important. The report section (page 9) of this guidance document should be carefully followed.

Compositing samples for verifying soil remediation is not acceptable without prior DNR approval. When verifying a soil remediation is complete, contaminant concentrations will be low. Compositing may result in the contaminant concentrations not being representative of what remains in the soil. If

concentrations are low, compositing may dilute the concentrations of a contaminant to below its threshold detection limit. Additionally, if contamination is indicated in a composited sample, the location of the contamination remains unknown.

Part 1 is divided into five main sections: Verifying Excavation Remedies, Verifying In-Situ and Ex-Situ Soil Remedies, Sample Analysis, Background Soil Samples, and Reports. The excavation and in-situ remediations require different strategies for verification. Guidance is presented for statistically determining background concentrations of compounds/contaminants. Guidance for reporting all appropriate information is presented to facilitate remediation approval.

- 2 - VERIFYING EXCAVATION REMEDIES

Verifying that contaminated soil is remediated by means of excavation requires samples from the excavation bottom and sidewalls. Tables and formulas presented provide the minimum number of samples necessary to verify cleanup for various size excavations. The biased approach specified in this guidance recommends soil sampling from areas most likely to still exceed cleanup criteria. The location of the sample collection points relies on site specific analysis of the release or contaminant distribution and the soil types encountered in the excavation. The minimum number of excavation floor and sidewall samples required to demonstrate verification using a point by point comparison with the cleanup criteria are specified. If the cleanup criteria are exceeded at any point, this verification methodology may require additional excavation at that point until the criteria are attained.

Sampling and analyzing the locations most likely to have contaminants can minimize the number of samples needed to verify remediation is complete. Since professional judgment and site specific knowledge are required for selecting sampling locations, the rationale used to select these locations must be documented in the verification report.

SAMPLE LOCATIONS

Using a biased sampling approach, samples must be collected where they will most likely encounter contamination which could exceed the cleanup criteria. This will minimize the number of samples needed to verify a site is remediated. A sampling strategy that uses bias to choose sample locations is recommended. While it is inappropriate for this guidance document to dictate exact locations for sample collection in this strategy, site specific information (e.g., the location of leaks in an underground storage tank or its piping) from the remedial investigation concerning the release and soil conditions should be used along with professional judgment and the general guidance provided here to select appropriate soil sampling locations.

EXAMPLE: It would be incorrect to sample the north side of an excavation pit as extensively as the south side when the leak was confirmed on the south side of the tank.

Because a site must be remediated to a certain degree before approval can be considered, an analysis of data generated by a prior investigation should yield information for the verification analysis. The field personnel present during remediation should be sufficiently familiar with the conditions on-site to implement an appropriate verification strategy. A soil verification strategy should incorporate all pertinent biases of a site which may include, but are not limited to, those listed below.

- preferential pathways of contaminant migration
- source areas
- stained soils

- other site specific "clues" (e.g., fractures in clays)
- changes in soil characteristics (e.g., sand/clay interfaces)
- soil types and characteristics

- 3 - NUMBER OF SAMPLES

The following tables are used to determine the minimum number of samples necessary from the floor and sidewalls of an excavation no greater than 0.25 acres using a biased sampling approach. If the area of the excavation floor exceeds 10,890 square feet, use Part 2 of this guidance document. A site may have an appropriate number of samples collected for verification, but, if the samples are not collected from the appropriate locations (discussed previously) and adequately reported, remediation may not be considered adequate. All sample locations must be accurately located, described, and reported. It should be noted that "excavation" as used here refers only to that area excavated for remediation purposes and being verified to meet Type A/Type B cleanup criteria.

Number of Excavation Floor Samples

Determine the minimum number of excavation floor samples from the table below.

TABLE 1 EXCAVATION FLOOR SAMPLES

Area of Floor (sq ft)	Number of Samples
< 500	2
500 < 1,000	3
1,000 < 1,500	4
1,500 < 2,500	5
2,500 < 4,000	6
4,000 < 6,000	7
6,000 < 8,500	8
8,500 <10,890	9

Number of Excavation Sidewall Samples

Sidewall samples are required to verify that the horizontal extent of contamination has been remediated. Use Table 2 to determine the minimum number of required sidewall samples. In no case is less than one sample on each sidewall (i.e., four) acceptable. In the case of irregularly shaped excavations, where four walls are not readily discernible, divide the total wall area into four segments of approximately equal size. Sidewall samples should be located in accordance with "biases" outlined earlier in Part 1.

- 4 -

TABLE 2 EXCAVATION SIDEWALL SAMPLES

Total Area of Sidewalls (sq ft)	Number of Samples
< 500	4
500 < 1,000	5
1,000 < 1,500	6
1,500 < 2,000	7
2,000 < 3,000	8
3,000 < 4,000	9
> 4,000	1 sample per 45 lineal feet of sidewall

VERIFYING IN-SITU AND EX-SITU SOIL REMEDIES

The effectiveness of in-situ soil remedies must be verified by three-dimensional random soil sampling. Refer to Attachment 2 for approved statistical sampling strategies. Certain ex-situ remedies, such as bio-piles or above-ground vapor extraction, may be amenable to statistical sampling strategies or batch sampling. Any proposed sampling strategy for in-situ or ex-situ remedies should be pre-approved by the DNR.

SAMPLE ANALYSIS

All test methods and associated target detection levels for cleanup verification must be consistent with those specified in MERA Operational Memorandum #6. Also, MERA Operational Memorandum #13 may be reviewed to evaluate appropriate QA/QC procedures. Generally, constituents in soil will be measured on a total, dry weight basis.

- 5 - BACKGROUND SOIL SAMPLES

ESTABLISHING SOIL BACKGROUND

Establishing soil background, as required by Act 307 PA 1982, as amended, Michigan Environmental Response Act (MERA), can be accomplished by utilizing Operational Memorandum #15 or using the following guidance.

Background should be established as appropriate for site specific waste constituents, specific chemicals used in various processes, facility operations, or remedial investigation results. Sample analyses may include metals, organic constituents, or other site specific waste constituents. Analyses should be in accordance with MERA Operational Memorandum #6

Many factors can play a part in the background concentrations of a chemical in soil.

EXAMPLE: The geologic origin (e.g., the parent rock) of glacial drift may have been high in copper, lead, or other metals that may be potential contaminants. Additionally, the hydrogeologic situation can alter the quantity of these elements. Groundwater recharge areas (e.g., highlands) are frequently leached of metals while groundwater discharge areas (e.g., swamps, floodplain) are the recipients of leached metals. Thus, sites in low areas will usually have higher background concentrations than upland areas. Other conditions, such as precipitation and atmospheric fallout from widely dispersed human and natural activities, also affect soil concentrations.

A minimum of four samples must be used to establish "background" in soils. This will help account for natural constituent occurrences and inherent variability within each distinctive soil horizon. Background samples must be collected in an area which has not been impacted by environmental contamination from the site and representative of natural background conditions. Based on waste type, contaminant mobility, operation practices, and soil type (sand, silty sand, clay), an estimate of contamination depth should be made and background samples taken at comparable depths for the particular soil type. Multiple soil horizons should have "background" established separately (e.g., minimum of four samples per each soil unit).

EXAMPLE:

```
    Ground Surface
Brown mediumcoarse SAND          4 samples
Lt. brown silty fine SAND        4 Samples
Gray silty CLAY w/trace of
```

fine-med sand 4 Samples

- 6 - STATISTICAL ANALYSIS FOR ESTABLISHING

BACKGROUND CONCENTRATIONS

The recommended statistical method for establishing background concentrations at small sites is (1) establishing the upper limit of background concentration of a constituent at the mean plus 3 standard deviations or (2) other statistical methods submitted to DNR for approval.

1. *Mean Plus 3 Standard Deviation Approach*

Calculate the "upper limit" of background concentration by using the following 5 step process.

1. A. Calculate the background mean (mb) by dividing the sum of the total background readings by the total number of background readings:

mb = (X1 + X2 + X3 + ... Xn) / n

B. Calculate the background variance (Sb2) by taking the sum of the squares of each reading minus the mean and dividing by the degrees of freedom (the total number of background samples minus one):

Sb2 =[(X2-mb)2+(X2-mb)2+...(Xn-mb)2] / n-1

NOTE: Any sample populations less than (n<30 samples) must use n - 1 for degrees of freedom

C. Calculate the background standard deviation (Sb) by taking the square root of the variance:

- 7 -

D. The Coefficient of Variation Test (CV) where

CV = - Sb / mb

is used to evaluate data distribution. The background data should generally have a CV of less than 0.5 for granular soils, less than 0.75 for cohesive soils, or an explanation accounting for higher CV values. The maximum recommended CV is 1. If the data distribution exceeds a CV of 1.0, then a thorough evaluation will need to be made to account for this variability (e.g., lab QA/QC, typographical errors, soil classification, sample location, data not normally distributed, etc.). If the CV exceeds 1.0 and there is sufficient evidence to suggest a data point does not accurately represent background conditions or if QA/QC problems exist which invalidate that data point, the outlier data may be dropped or additional samples collected and analyzed to ensure a sufficient representative data population (n) is achieved. A high concentration in and of itself is not sufficient justification to exclude the data point.

E. Use the + 3*Sb of "background" data as the maximum allowable limit or upper limit. Where 3*Sb equals three times the standard deviation and equals the background mean (this statistical method only requires one sample per station). Compare each sample point to the calculated maximum allowable limit or upper limit analyzed from background data.

EXAMPLE: Four sand samples from a site were analyzed for background concentrations for lead. Concentrations of lead from the sample analyses returned from the lab were 56, 25, 18, and 35 ppb. Now, the investigator wants to examine the data set to discover whether the 56 ppb sample is an outlier:

mb = (56 + 25 + 18 + 35) / 4 = 33.5

Sb2 = ([56-33.5]2+[25-33.5]2+[18-33.5]2+[35-33.5]2) / 3

Sb2 = 273.67

Sb = 16.5 (Standard Deviation)

CV = 16.5 / 33.5 = 0.49

Because 0.49 is less than 0.5, no further evaluation of the background data set is necessary.

Therefore, the background upper limit value for this site is

Background Upper Limit = mb + (3*Sb) = 33.5 + (3*16.5) = 83.0 ppb

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If a value is found to be an outlier which is not representative of background conditions, it may be replaced by another sample that is not an outlier to maintain at least four samples for background determination.

- 8 -

2. *Other statistical procedures for establishing background.* Refer to a statistical reference book or US EPA's Interim Final Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities (April 1989) and Addendum (July 1992).

PROCEDURES FOR NON-DETECT VALUES

The following provides some guidelines in incorporating non-detectable sample results into the procedure to calculate background concentrations.

1. If less than 50% of the background data is below the detection limit (DL), use ½ of the detection limit as the value.

2. If more than 50% of the background data is below the detection limit, use one of the following procedures.

- Alternate "O" and the detection limit (DL) resulting in a net value of of the detection limit with a variance.

EXAMPLE:	Actual Value	Substitute Value
	<DL	DL
	<DL	0
	<DL	DL
	<DL	0

- The Continuity Correction procedure with the t-test, Cohen's method, or other approved methods.

- 9 - REPORT FOR SMALL SITE VERIFICATION

Soil cleanup verification reports for small sites must identify the number and location of samples and justify the sample location selected (why and how). The verification report must include the following.

1. MAP(s) and CROSS SECTIONS

Provide a scaled map of the floor and walls of an excavation (the vertical and horizontal area treated for in-situ remediations) with sample locations identified. The cross section should depict the stratigraphy, fractures, soil types, discolorations, unusual characteristics, odor, etc.

2. SAMPLE LOCATION RATIONALE

A. Background sample locations

B. Verification sample locations

C. Sample depths

D. Sample collection procedures

E. Describe biases and rationale used for collecting each sample (e.g., clay fractures, discolored soil, location of leak in tank)

3. DATA ANALYSES

A. Analytical parameters

B. Analytical methods used

C. Method detection limits

D. Laboratory Quality Assurance/Quality Control

4. STATISTICAL ANALYSES

A. Calculation of background concentrations

B. Coefficient of variance calculations

C. Lab results

D. Narrative explanation of background concentrations

- 10 - DNR--GUIDANCE DOCUMENT, PART 2

MEDIUM AND LARGE SITE SOIL CLEANUP VERIFICATION

(GREATER THAN 10,890 SQUARE FEET)

Part 2 describes statistical random sampling strategies to verify the remediation of medium and large sites greater than 0.25 acres in size. The strategies employ the use of gridding to facilitate the

unbiased selection of sampling points and accepted statistical tools for evaluating the resultant data. The strategies provide a 95% confidence level of determining any hot spot concentrations on a site. It contains guidance on sampling protocol and necessary documentation for clean closures. Part 2 also discusses how to establish grid intervals, set grids, sample grids, statistically evaluate the data, use grids to guide additional remedial activities, disposal options, reporting, and a certification checklist. It also provides guidance on the sampling of ex-situ remedial processes (e.g., thermal desorption).

The term `clean closure' means that the site has been restored to either Type A or Type B levels. Type A is defined in Act 307 P.A. 1982, as amended, which references nondetect or background levels. Type B is defined in Act 307 P.A. 1982, as amended, which references riskbased or background levels. Waste, soil, other environmental media, and/or debris removed should be classified as hazardous or non-hazardous to determine disposal options and handling requirements (i.e., solid waste under Act 641 P.A. 1978, as amended; hazardous waste under Act 64 P.A. 1979, as amended; land ban restrictions under 40 CFR Part 268).

All cleanup verification evaluations must consider the spatial arrangements of sample values (patterned vs totally random) and the impacts on the present and future uses of the site. Because Type B cleanups are based on residual risk, the distribution of that risk, now and in the future, must be determined. These procedures are **not *absolutes***. Other sampling approaches may be developed and submitted for DNR approval.

Three of the statistical sampling strategies most commonly used for evaluating remedial sites and wastes are described in Attachment 2. For further discussion on sampling strategies and sample collection methods, see "Test Methods for Evaluating Solid Waste," SW-846 Volume II: Field Methods, November 1986, Third Edition, US EPA.

Compositing samples for verifying soil remediation is not acceptable without prior DNR approval. When verifying a soil remediation is complete, contaminant concentrations will be low. Compositing may result in the contaminant concentrations not being representative of what remains in the soil. If concentrations are low, compositing may dilute the concentrations of a contaminant to below its threshold detection limit. Additionally, if contamination is indicated in a composited sample, the location of the contamination remains unknown.

- 11 - ESTABLISHING GRID INTERVALS

When obtaining samples to verify that soil or wastes have been adequately remediated, it is important to insure that the analytical results obtained will provide an accurate representation of the entire area or volume under consideration. The location and number of samples to be taken at a particular remediation site depends on many factors: the level of confidence desired, the spatial and temporal variability of the media to be sampled, and the costs involved. An important objective in any sampling program is to obtain the most accurate data possible while minimizing the associated costs. One method to accomplish this goal is to use statistically valid sampling strategies. The appropriate sample number can be estimated and the sampling locations can be chosen without bias.

Such strategies employ the use of gridding to facilitate the unbiased selection of sampling points and accepted statistical tools for evaluating the resultant data. Statistical theory allows for the sampling of a subset of the grid points to achieve a reliable characterization of the entire remedial area or waste. Subsections describe ways to use sampling grids and statistical tools to evaluate areas of remediation.

The following equations and tables provide a simple basis to establish a grid system to facilitate

unbiased selection of sampling points and sample coverage proportional to the area being verified.

1. *Basic Strategies.* A grid system should be established over the area being remediated. Grid point representation should be proportional to the size of the area. For excavation, both the sidewalls and bottom areas would be included in the determination of the area size. It is recommended that one of the following equations be used to determine grid intervals for stationing:

small site: see Part 1

square root

medium site: GI = (A/pi) 1/2 / 4

large site: GI =(Api/SF) 1/2

```
WHERE:    a  = area to be grid (square feet)
          GI  = grid interval
          SF  =  Site Factor, length of area to be grid (unitless)
```

- 12 -

It appears that there are logical size ranges of sites to which the grid equations apply:

A) small: up to 0.25 acre

B) medium: 0.25 3.0 acres

C) large: 3.0 acres and greater

To simplify this application, use the following chart based on an average size range of sites (1 acre = 43,560 square feet). The approximate grid ranges are provided as a quick check on numbers generated for specific sites using the above formulas.

Site Acreage*	Square Feet*	Grid Interval Ranges
up to 0.25 (small)	up to 10,890	See Part 1
0.25-3.00 (medium)	10,890-130,680	15-50 feet
3.0 and over (large)	130,680 +	30 feet plus

* Site acreage, square footage, is total area of sidewalls and base of excavation.

2. *Setting the Grid.* After the grid interval is calculated, it is recommended that a scaled grid overlay be made to superimpose on a map of the remediated area (this area includes both sidewalls and base). Some specified point (usually the southwest corner) should be designated as the 0,0 coordinate. The grid can then be adjusted to maximize sampling coverage. Some grid adjustment may be necessary for unusually shaped areas. Grid adjustment may also be needed to accommodate a minimum of at least one sample from each sidewall. Proposals for different grid strategies may be submitted for DNR review and approval on a case-by-case basis.

- 13 -

3. ***Variations on Basic Strategy.***

A. Subgridding. It may be warranted to apply grids with different intervals within the remediated area so that a proportional sampling can be focused on suspect areas (such as sumps, tank leak areas, etc.).

B. Further Randomization. Sites that may have a patterned distribution of waste or contamination due to time sequence of filling, production sequences, or physical site conditions (i.e., furrows) may require a further randomization of sampling. In such cases, the following grid cell sampling format may be selected instead of at grid point stations. Each grid cell to be sampled must be divided into nine equal sized "subcells." Next, a random number table is used to select in which of the subcells the sample will be taken. The random number table is used again to select which subcell for the next cell and so on.

In the example, a sampling grid has been set up with grid point stations 20 feet apart using the appropriate formula. Two cells which have been selected at random have been divided into nine subcells each. Subcell #4 was chosen randomly in one cell and subcell #2 in the other cell. This process is continued for all of the cells selected at random for sampling. Samples are then taken in each randomly chosen subcell.

- 14 -

C. Three dimensional gridding: In-Situ and Ex-Situ Remediations.

In-situ and ex-situ remediations involving soils and/or wastes with a significant vertical component should be evaluated in three dimensions (volume evaluation). Examples of such remediations would be in-situ soil vapor extraction or ex-situ bioremediation involving several cubic feet of soil and/or waste. A grid is superimposed on the remediation area as described in the previous sections and a vertical component is added at each node. The vertical sampling increments would be site specific and require prior approval from the DNR. Refer to Attachment 1 "Guide to Sample Bias" for additional guidance on vertical sampling increments.

SAMPLING OF GRID

Sampling of grids may include all of the grid stations or a phased subset of the total stations. The subset of grid stations is created by assigning coordinates to all the nodes and randomly selecting nodes using a random number generator or a random number table (refer to Attachment 2). A minimum of 12 samples or 25%, whichever is larger, of the total grid stations should be sampled and analyzed initially to allow a large enough data pool for statistical analysis. It is advisable that extra samples also be taken and kept under proper chain of custody and storage procedures at the time of initial sampling. If the statistical analysis indicates that more samples are needed, an additional sample trip to the field may have been avoided. A method for calculating the sample size requirements is presented in Attachment 2 (Lamda relationship).

ESTABLISHING SOIL BACKGROUND

Establishing soil background, as required by Act 307 PA 1982, as amended, Michigan Environmental Response Act (MERA), can be accomplished by utilizing Operational Memorandum #15 or using the following guidance.

Background should be established for site specific waste constituents, specific chemicals used in various processes, facility operations, or remedial investigation results. Sample analyses may include metals, organic constituents, or other site specific waste constituents. Analyses should be in accordance with Act 307 P.A. 1982, as amended.

Many factors can play a part in the background concentrations of a chemical in soil.

EXAMPLE: The geologic origin (e.g., the parent rock) of glacial drift may have been high in copper, lead, or other metals that may be potential contaminants. Additionally, the hydrogeologic situation can alter the quantity of these elements. Groundwater recharge areas (e.g., highlands) are frequently leached of metals while groundwater discharge areas (e.g., swamps, floodplain) are the recipients of leached metals. Thus, sites in low areas will usually have higher background concentrations than upland areas. Other conditions, such as precipitation and atmospheric fallout from widely dispersed human and natural activities, also affect soil concentrations.

- 15 -

A minimum of four samples must be used to establish "background" in soils. This will help account for natural constituent occurrences and inherent variability within each distinctive soil horizon. Background samples must be collected in an area which has not been impacted by environmental contamination from the site and representative of natural background conditions. Based on waste type, contaminant mobility, operation practices, and soil type (sand, silty sand, clay), an estimate of contamination depth should be made and background samples taken at comparable depths for the particular soil type. Multiple soil horizons should have "background" established separately (e.g., minimum of four samples per each soil unit).

EXAMPLE:

Ground Surface	
Brown medium-coarse SAND	4 samples
Lt. brown silty fine SAND	4 samples
Gray silty CLAY w/trace of fine-med sand	4 samples

STATISTICAL ANALYSIS FOR ESTABLISHING BACKGROUND CONCENTRATIONS

The recommended statistical method(s) for establishing background concentrations at medium and large sites are (1) establishing the upper limit of background concentration of a constituent at the mean plus 3 standard deviations, (2) tolerance limit, (3) t-tests, and (4) other statistical methods submitted to the DNR for approval.

1. *Mean Plus 3 Standard Deviation Approach.*

Calculate the "upper limit" of background concentration by using the following 5 step process.

A. Calculate the background mean () by dividing the sum of the total background readings by the total number of background readings:

$m = (X1 + X2 + X3 + \ldots Xn) / n$

- 16 -

B. Calculate the background variance (Sb2) by taking the sum of the squares of each reading minus the mean and dividing by the degrees of freedom (the total number of background samples minus one):

Sb2 = [(X2-m)2+(X2-m)2+...(Xn-m)2] / n-1

NOTE: Any sample populations less than (n<30 samples) must use n - 1 for degrees of freedom

C. Calculate the background standard deviation (Sb) by taking the square root of the variance:

D. The Coefficient of Variation Test (CV) where

CV = Sb / m

is used to evaluate data distribution. The background data should generally have a CV of less than 0.5 for granular soils, less than 0.75 for cohesive soils, or an explanation accounting for higher CV values. The maximum recommended CV is 1. If the data distribution exceeds a CV of 1.0, then a thorough evaluation will need to be made to account for this variability (e.g., lab QA/QC, typographical errors, soil classification, sample location, data not normally distributed etc.). If the CV exceeds 1.0 and there is sufficient evidence to suggest a data point does not accurately represent background conditions or if QA/QC problems exist which invalidate that data point, the outlier data may be dropped or additional samples collected and analyzed to ensure a sufficient representative data population (n) is achieved. A high concentration in and of itself is not sufficient justification to exclude the data point.

- 17 -

E. Use the + 3*Sb of "background" data as the maximum allowable limit or upper limit. Where 3*Sb equals three times the standard deviation and equals the background mean (this statistical method only requires one sample per station). Compare each sample point to the calculated maximum allowable limit or upper limit analyzed from background data.

EXAMPLE: Four sand samples from a site were analyzed for background concentrations for lead. Concentrations of lead from the sample analyses returned from the lab were 56, 25, 18, and 35 ppb. Now, the investigator wants to examine the data set to discover whether the 56 ppb sample is an outlier:

m = (56 + 25 + 18 + 35) / 4 = 33.5

Sb2 = ([56-33.5]2+[25-33.5]2+[18-33.5]2+[35-33.5]2) / 3

Sb2 = 273.67

Sb = 16.5 (Standard Deviation)

CV = 16.5 / 33.5 = 0.49

Because 0.49 is less than 0.5, no further evaluation of the background data set is necessary.

Therefore, the background upper limit value for this site is

Background Upper Limit = m + 3*Sb

Background Upper Limit = 33.5 + (3*16.5)

Background Upper Limit = 83.0 ppb

If a value is found to be an outlier which is not representative of background conditions, it may be replaced by another sample that is not an outlier to maintain at least four samples for background determination.

2. *Tolerance Limit.* This statistical procedure is a fairly sensitive program for environmental purposes. It minimizes false positive and is simple to perform. **A minimum background data base of n=8** (optimum n=16) is needed for this method. Other suggested criteria follow:

A. The Coefficient of Variation Test (CV) to evaluate data distribution. See this Guidance Document, Part 2, Statistical Analysis for Establishing Background Concentrations, #1.D. (the Coefficient of Variation Test....).

B. Using the mean () and standard deviation (Sb), construct the onesided upper tolerance limit (TL) by taking the mean plus a tolerance coefficient (K) at the 95% probability level for a 95% coverage (for K values, see Attachment 3) times the standard deviation as follows:

TL = m + KSb

- 18 -

3. *ttests.* Any ttest should be "approved" by DNR prior to use since there are a number of variations. The Gosset Student ttest (1908) or Cochran's Approximation to the Behren'sFisher Student's ttest as referenced in the 40 CFR Part 264, Appendix IV, are recommended. **Note** that these statistical comparison methods require that two or more discrete samples be taken at each sampling station.

4. *Other statistical procedures for establishing background.* Refer to a statistical reference book or US EPA's Interim Final Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities (April 1989) and Addendum (July 1992).

PROCEDURES FOR NON-DETECT VALUES

The following provides some guidelines in incorporating non-detectable sample results into the procedure to calculate background concentrations.

1. If less than 50% of the background data is below the detection limit (DL), use ½ of the detection limit as the value.

2. If more than 50% of the background data is below the detection limit, use one of the following procedures.

Alternate "O" and the detection limit (DL) resulting in a net value of of the detection limit, with a variance

EXAMPLE:	Actual Value	Substitute Value
	<DL	DL
	<DL	0
	<DL	DL
	<DL	0

The Continuity Correction procedure with the t-test, Cohen's method, or other approved methods.

STATISTICAL EVALUATION OF DATA

A detailed description of an acceptable approach for evaluating the data generated by statistically based random sampling strategies such as those described in the foregoing sections is provided in Attachment 2 (page 29). The 95% upper confidence limit (UCL) of the mean is calculated for each constituent of concern and compared to the regulatory threshold (RT) (i.e., cleanup criterion; e.g., Type A or B). If the UCL is less than the RT and an adequate number of samples have been collected and spatially evaluated, the remediation is deemed complete. Attachment 2 also provides a step wise procedure for determining whether an adequate number of samples have been collected, based on the analytical data derived from the initial and subsequent rounds of samples. **All evaluations must consider the spatial correlation of sample values** (e.g., highest concentrations in the same area), **present and future uses of the site, residual risk, and distribution of that risk now and in the future.** Other acceptable methods for UCL and sample size calculations can be found in US EPA SW-846, Third Edition, Section 9.1.1.3.

- 19 - GRID APPROACH TO ADDITIONAL REMEDIATION

1. *TwoDimensional Node Sampling Excavation Grid.* Verification sampling as described above will at times indicate that remediation is incomplete. Excavation of contaminated areas should be based on the established grid system interval (as recommended in this Guidance Document, Part 2). Where a subset of grid points has indicated that the entire area exceeds the cleanup, the nodes adjacent to the sampled nodes that are causing the exceedance should be sampled, and this process repeated until the "Hot spots" requiring removal have been defined. The radius of excavation around the contaminated sample point(s) is equal to the grid interval (GI=r). Excavation depth is to the deepest point of contamination or to the depth where acceptable levels are anticipated. After excavation, the impacted point(s) must be resampled at their new elevations to verify that the area meets the selected cleanup criteria. If continued contamination is detected, the excavation format is repeated until a satisfactory result is obtained.

Remediation of contaminated soil by excavation will be in accordance with Act 307 P.A. 1982, as amended. The proposed remedial action plan must be approved by the DNR.

2. *Two-Dimensional Subcell Sampling Excavation Grid.* Use this Guidance Document, Part 2. The radius of excavation around a contaminated point may need to be adjusted to greater than the GI distance. This adjustment is due to the variable distances between sampling points.

3. *ThreeDimensional Cleanup Verification.* If sampling and statistical analysis using this Guidance Document indicate that Act 307 cleanup criteria have not been met, additional remediation will be required. The sampling protocol and strategies described in Attachment 2 and in SW-846, Third Edition, Volume II, Part III, Chapter 9, are acceptable. All sampling strategies, detection levels, and sampling pathways must be in accordance with Act 307 P.A. 1982, as amended. If any portion of the soil mass in question appears to be causing the material to fail, it may be identified through hot spot sampling and selectively removed. Subsequent sampling must be done to confirm that the remaining material meets Act 307 P.A. 1982, as amended.

4. *Batch Sampling for exsitu treatment processes.* If exsitu treatment processes of contaminated soil or waste is used in the remediation, a sampling program for the process stream needs to be developed. The basis of this program is to get representative samples over time versus a spatial approach (Attachment 2, Sampling Process Streams).

- 20 - DISPOSAL OPTIONS

Soils remediated to Act 307 P.A. 1982, as amended, standards (Type A and/or Type B) are no longer considered a waste per Act 64 P.A. of 1979, as amended, and RCRA regulations. Disposal of excavated waste, soil, other environmental media, and/or debris must be in accordance with all applicable Federal, State, and local regulations.

REPORT FOR MEDIUM AND LARGE SITES VERIFICATION

Soil cleanup verification reports for medium and large sites must identify the number and location of samples and justify the sample location selected (why and how). The verification report must include the following.

1. MAP(S) AND CROSS SECTIONS

Provide a scaled map of the floor and walls of the excavation (the vertical and horizontal area treated for in-situ remediations) with sample grid and sample locations identified. Appropriate cross section should depict the stratigraphy, fractures, soil types, and final depth and elevations of the excavation.

2. SAMPLE LOCATION RATIONALE

a. Properly labeled and easily identified sampling grid stations (map) including background stations

b. Sample Depths

c. Sample Collection Procedures

d. Results of all tests to determine clean closure (charts, tables, lab sheets, field notes, well logs, boring logs)

3. DATA ANALYSES

a. Analytical parameters

b. Analytical methods used

c. Method detection limits

d. Laboratory Quality Assurance/Quality Control

4. STATISTICAL ANALYSES

a. Explanation and calculation of background concentrations

b. Statistical comparisons on sampling results compared to background (this should include full computations on background and statistical analysis)

c. Lab results

5. Additional information to support closure (e.g., residual risks, spatial correlation of sample values, present and future land uses)

RCRA CLEAN CLOSURE CERTIFICATION CHECKLIST

Attachment 4 is a guide that indicates the information that a facility should provide to certify that their activities meet the conditions for a clean closure under the Act 64/RCRA regulations.

- 21 - A T T A C H M E N T 1

GUIDE TO SAMPLE BIAS

Many factors can play a part in the concentrations of contaminants. The following contains some of the factors impacting chemical concentrations and locations.

CHEMICAL TRANSFORMATIONS

Many organic chemicals may undergo aerobic and anaerobic degradation. A description of these processes is beyond the scope of this document. The subject is approached here, however, to be sure that samplers are aware that the chemical(s) spilled may not be the only chemical(s) in the soil after a transformation has occurred. These occurrences should be documented in the remedial investigation. The full scan of chemicals from the remedial investigation requiring cleanup should be analyzed when doing a closure. Analyses should be done for all chemicals that have been identified as breakdown products of the chemicals found on-site.

The professional literature contains many articles on this subject (Cline and Brown, 1989; Borden and Bedient, 1987; Wilson and Wilson, 1985). The interested reader is directed to these articles.

Organic Carbon Content of Soil

The organic carbon content of soils is a key factor in the ability of any soil to adsorb contaminants. For a variety of reasons (Lindsay, 1979), an increase in organic carbon content leads to an increase in the adsorption of several classes of chemicals.

Where to sample: Areas of the excavation that appear to have excess organic carbon (e.g. peat, muck, darker soils) should be preferentially sampled.

Medium Sand or Larger Grains

Medium to larger grain size sand has from 20 to 40 percent porosity. Most sands in Michigan are composed of quartz, limestone, and small amounts of metamorphic rock fragments. These soils have a low capacity for adsorbing metals or hydrophilic (soluble) organic chemicals. Hydrophobic (insoluble) organic chemicals with low molecular weight will adsorb to this soil in small amounts. Hydrophobic chemicals with high molecular weight will adsorb in moderate amounts (Cline & Brown, 1989). These soils have a low capacity to hold contaminants in the grain interstices due to low capillary action. Contaminants that are held in these soils adhere to the grains themselves in dry soils and are forced into the smaller pore spaces in wet soils (Schwille, 1988).

Where to sample: Samples should be placed at regular intervals along the base and sidewalls of the excavation being sure that samples are located where the source was removed. In these soils, the capillary force is low enough to ignore its effects in transporting contaminants lateral to gravity. Therefore, sidewall samples should be located near the excavation floor. This is especially true for low surface tension products such as gasoline.

The limestone sand grains can act as a buffer to contaminants that cause pH changes (e.g., steel mill pickling acids). For these types of contaminants, the sampler should be on the lookout for

intra-granular precipitates. These can appear as grain surface staining or make the soil appear clumpy or aggregated. Soils containing precipitates should be sampled.

- 22 -

Fine Sand and Silt

These soils have strong capillary action due to the small inter-granular distances. A determination of the fluid surface tension of the spilled product is helpful. High surface tension aids in the ability of a substance to overcome gravity by capillary action. As before, higher molecular weight products can be expected to adsorb to the grains to a greater degree. This allows a product to move lateral to gravity and, to a degree, upward from the leak location. Low surface tension products, such as TCE (trichloroethene), are more likely to go straight down than oils in these kinds of soils. However, the hydraulic head (i.e., the amount of product in the original spill) must be substantial to force a dense non-aqueous phase liquid through a media with a hydraulic conductivity less than 1 x 10-3 cm/sec (Schwille, 1988).

Where to sample: Interfaces between fine sand layers with larger grains above should be sampled. When high surface tension contaminants are suspected, silt layers should be sampled.

Clays

Clay soils are very different from the sands and silts. Clays possess a net negative charge. This causes heavy metal cations (e.g., Cr+6, Cd+2, Pb+2) to adsorb to the clay surface. In fact, this is true for any positive ionizable substance. Clays also have a much greater secondary porosity than primary (primary porosity is the space between the soil particles; secondary porosity is the space between fractures, bedding planes, and soil structures). As a result, spills in clay soils tend to follow preferred pathways. Clays will often show signs of shrinkage cracks or fractures that will allow contaminants to migrate in what would otherwise be considered a "tight" soil in a lab analysis of permeability. Signs of fracturing include "patterned" mottling. This is where the Fe (and also Mn) will be oxidized to a red, yellow, or reddish brown color along the crack while the matrix remains the reduced blue/gray color (Lindsay, 1979).

Where to sample: It is very important to take clay soil samples from fractures. The fractures are the avenue of travel for contaminants in clay soils. Clay soils may also have sand lenses which should always be sampled. Sand lenses in clays tend to collect fluids. As such, they may harbor contaminants.

Bedrock

Excavations in bedrock present difficult problems. Unlike clay, some bedrock formations have substantial primary porosity as well as secondary porosity. In Michigan, these are sandstones, conglomerates, and brecciated/coarse grained limestones. Examples of bedrock in Michigan with low primary porosity are fine grained limestones, shale, and crystalline metamorphic rocks (e.g., gneiss). If the sampler is unaware of the type of bedrock that is in an excavation, a geologist must be consulted.

Where to sample: Excavations in areas of bedrock with significant primary porosity must be sampled in both the fractures and the matrix. Bedrock without primary porosity should have sampling predominantly in the fractures as in the clay situation. Weathered zones in bedrock will hold contaminants better than unweathered zones. This is due to the increased number of adsorption sites available in weathered rock.

- 23 - A T T A C H M E N T 2

SAMPLING PROTOCOL

FOR CHARACTERIZING WASTE/TREATMENT LEVELS:

STRATEGIES FOR EVALUATING TREATED SOILS AND WASTE MATERIALS

When obtaining samples to characterize a treated soil or waste material, it is important to insure that the analytical results obtained will provide an accurate estimation of the nature of the entire area/volume under consideration. The location and number of samples to be taken at a particular site depend on many factors: the degree of accuracy desired, the spatial and temporal variability of the media to be sampled, and the costs involved. An important objective in any sampling program is to obtain the most accurate data possible while minimizing the associated costs. One method to accomplish this goal is to use statistically valid sampling strategies. The appropriate sample number can be estimated and the sampling locations can be chosen without bias.

Attachment 2 provides information on the methods used to obtain accurate data while minimizing the costs. The attachments include a discussion of three statistical sampling strategies and methods to determine the appropriate grid size for the area under investigation. If several areas on a site are under investigation, it may be advisable to grid them separately. This is especially true if information does not exist to indicate that the areas contain similar constituents or that they were placed at the same time period.

Information is also supplied on the statistical evaluation of the resultant analytical data. A minimum of 12 samples or 25%, whichever is greater, of the total grid stations should be sampled and analyzed initially to allow a large enough data pool for the statistical analysis. Extra samples should be taken and kept under proper chain of custody and handling procedures at the time of initial sampling. If the statistical analysis indicates that two or three more samples are needed, an additional trip to the field may not be necessary. This may also avoid the need to reestablish the grid pattern at a later date.

For further discussion on sampling strategies and sample collection methods, see "Test Methods for Evaluating Solid Waste," SW846 Volume II: Field Methods, November 1986, Third Edition, US EPA.

- 24 - STATISTICAL SAMPLING STRATEGIES

Statistical sampling strategies can often produce increased data accuracy while eliminating sampler bias. Random sampling is based on the theory of random chance probabilities in order to choose the most representative sample. Knowledge of the waste distribution is not necessary. The error in data accuracy of a random sampling scheme can be objectively measured since the probability of choosing each sampling point is known. A random numbers table (attached) or a random numbers generator should be used to select the sampling locations eliminating bias by the sample collector.

Several statistical sampling strategies are available to produce an unbiased, representative sampling program. The principles behind three of these and the situations for which they are best suited are provided below. To achieve true random sampling, composite sampling is not acceptable.

1. ***Simple Random*** is a method that requires little or no prior knowledge of material distribution. It relies on random chance probability theory--where each sampling location has an equal and known probability of being selected. In this way, sampling error can be accurately estimated. Usually, the

area of interest is sectioned into a two or three dimensional grid pattern and random coordinates are chosen for sampling.

2. ***Systematic random*** is an extension of simple random sampling that may produce a more efficient sampling survey. It can be more efficient by reducing the sampling error while maintaining the sample number, or by reducing the number of samples needed to achieve a specified sampling error, or by reducing the cost of collection. This method also requires little or no knowledge about the waste distribution, but bias and imprecision can be introduced if unseen trends or cycles exist. Two methods used to select sample locations under this method follow.

A) randomly selecting a transect or transects and sampling at preselected intervals

B) preselecting both the transect or transects and the sampling interval and starting from a randomly selected point.

3. ***Stratified random sampling*** requires some knowledge about the waste distribution. When stratification is known or suspected, sampling efficiency can be improved by dividing the material into strata that are more homogeneous than the total area. Simple random sampling techniques can then be used to sample each stratum independently. Each stratum is divided into a grid pattern and the sampling points are selected randomly. If the area is vertically stratified, the sampling points in each stratum are selected randomly and then selected depths are sampled. If the area is horizontally stratified, the sampling points within each stratum are selected randomly, but the total depth is sampled. An analysis of variance (ANOVA) should be done on the analytical results to determine if the strata differ significantly. This is done to assure that the use of stratified random sampling was statistically valid. When the volume of the strata differ or the number of samples within each strata differs, the results must be weighed appropriately to avoid bias.

- 25 - RANDOM NUMBERS TABLE

HOW TO USE THE RANDOM NUMBERS TABLE

1. Determine the number of samples you need to take. Identify the number of digits necessary to cover the sample population (e.g., for a sample population of 55, two digits are necessary to cover the selected grid stations 01 through 55).

2. Using the random numbers table, choose any number as a starting point.

3. From this starting point number, go in any direction and continue in the same direction and pattern sequence until you have selected the predetermined number of samples with no repetitions. Numbers larger than the population size are ineligible (e.g., numbers greater than 55 in the example are ineligible).

Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(
1	10480	15011	01536	02011	81647	91646	69179	14194	62590	36207	20
2	22368	46573	25595	85393	30995	89198	27982	53402	93965	34095	52
3	24130	48360	22527	97265	76393	64809	15179	24830	49340	32081	30
4	42167	93093	06243	61680	07856	16376	39440	53537	71341	57004	00
5	37570	39975	81837	16656	06121	91782	60468	81305	49684	60672	14
6	77921	06907	11008	42751	27756	53498	18602	70659	90655	15053	21
7	99562	72905	56420	69994	98872	31016	71194	18738	44013	48840	63
8	96301	91977	05463	07972	18876	20922	94595	56869	69014	60045	18
9	89579	14342	63661	10281	17453	18103	57740	84378	25331	12566	58

10	85475	36857	43342	53988	53060	59533	38867	62300	08158	17983	16
11	28918	69578	88231	33276	70997	79936	56865	05859	90106	31595	01
12	63553	40961	48235	03427	49626	69445	18663	72695	52180	20847	12
13	09429	93969	52636	92737	88974	33488	36320	17617	30015	08272	84
14	10365	61129	87529	85689	48237	52267	67689	93394	01511	26358	85
15	07119	97336	71048	08178	77233	13916	47564	81056	97735	85977	29
16	51085	12765	51821	51259	77452	16308	60756	92144	49442	53900	70
17	02368	21382	52404	60268	89368	19885	55322	44819	01188	65255	64
18	01011	54092	33362	94904	31273	04146	18594	29852	71585	85030	51
19	52162	53916	46369	58586	23216	14513	83149	98736	23495	64350	94
20	07056	97628	33787	09998	42698	06691	76988	13602	51851	46104	88
21	48663	91245	85828	14346	09172	30168	90229	04734	59193	22178	30
22	54164	58492	22421	74103	47070	25306	76468	26384	58151	06646	21
23	32639	32363	05597	24200	13363	38005	94342	28728	35806	06912	17
24	29334	27001	87637	87308	58731	00256	45834	15398	46557	41135	10
25	02488	33062	28834	07351	19731	92420	60952	61280	50001	67658	32
Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(
26	81525	72295	04839	96423	24878	82651	66566	14778	76797	14780	13
27	29676	20591	68086	26432	46901	20849	89768	81536	86645	12659	92
28	00742	57392	39064	66432	84673	40027	32832	61362	98947	96067	64
29	05366	04213	25669	26422	44407	44048	37937	63904	45766	66134	75
30	91921	26418	64117	94305	26766	25940	39972	22209	71500	64568	91
31	00582	04711	87917	77341	42206	35126	74087	99547	81817	42607	43
32	00725	69884	62797	56170	86324	88072	76222	36086	84637	93161	76
33	69011	65797	95876	55293	18988	27354	26575	08625	40801	59920	29
34	25976	57948	29888	88604	67917	48708	18912	82271	65424	69774	33
35	09763	83473	73577	12908	30883	18317	28290	35797	05998	41688	34
36	91567	42595	27958	30134	04024	86385	29880	99730	55536	84855	29
37	17955	56349	90999	49127	20044	59931	06115	20542	18059	02008	73
38	46503	18584	18845	49618	02304	51038	20655	58727	28168	15475	56
39	92157	89634	94824	78171	84610	82834	09922	25417	44137	48413	25
40	14577	62765	35605	81263	39667	47358	56873	56307	61607	49518	89
41	98427	07523	33362	64270	01638	92477	66969	98420	04880	45585	46
42	34914	63976	88720	82765	34476	17032	87589	40836	32427	70002	70
43	70060	28277	39475	46473	23219	53416	94970	25832	69975	94884	19
44	53976	54914	06990	67245	68350	82948	11398	42878	80287	88267	47
45	76072	29515	40980	07391	58745	25774	22987	80059	39911	96189	41
46	90725	52210	83974	29992	65831	38857	50490	83765	55657	14361	31
47	64364	67412	33339	31926	14883	24413	59744	92351	97473	89286	35
48	08962	00358	31662	25388	61642	34072	81249	35648	56891	69352	48
49	95012	68379	93526	70765	10593	04542	76463	54328	02349	17247	28
50	15664	10493	20492	38391	91132	21999	59516	81652	27195	48223	46
51	16408	81899	04153	53381	79401	21438	83035	92350	36693	31238	59
52	18629	81953	05520	91962	04739	13092	97662	24822	94730	06496	35
53	73115	35101	47498	87637	99016	71060	88824	71013	18735	20286	23
54	57491	16703	23167	49323	45021	33132	12544	41035	80780	45393	44
55	30405	83946	23792	14422	15059	45799	22716	19792	09983	74353	68
56	16631	35006	85900	98275	32388	52390	16815	69298	82732	38480	73
57	96773	20206	42559	78985	05300	22164	24369	54224	35083	19687	11
58	38935	64202	14349	82674	66523	44133	00697	35552	35970	19124	63
59	31624	76384	17403	53363	44167	64486	64758	75366	76554	31601	12
60	78919	19474	23632	27889	47914	02584	37680	20801	72152	39339	34
61	03931	33309	57047	74211	63445	17361	62825	39908	05607	91284	68
62	74426	33278	43972	10119	89917	15665	52872	73823	73144	88662	88
63	09066	00903	20795	95452	92648	45454	09552	88815	16553	51125	79
64	42238	12426	87025	14267	20979	04508	64535	31355	86064	29472	47
65	16153	08002	26504	41744	81959	65642	74240	56302	00033	67107	77

66	21457	40742	29820	96783	29400	21840	15035	34537	33310	06116	95
67	21581	57802	02050	89728	17937	37621	47075	42080	97403	48626	68
68	55612	78095	83197	33732	05810	24813	86902	60397	16489	03264	88
69	44657	66999	99324	51281	84463	60563	79312	93454	68876	25471	93
70	91340	84979	46949	81973	37949	61023	43997	15263	80644	43942	89
71	91227	21199	31935	27022	84067	05462	35216	14486	29891	68607	41
72	50001	38140	66321	19924	72163	09538	12151	06878	91903	18749	34
73	65390	05224	72958	28609	81406	39147	25549	48542	42627	45233	57
74	27504	96131	83944	41575	10573	08619	64482	73923	36152	05184	94
75	37169	94851	39117	89632	00959	16487	65536	49071	39782	17095	02
Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(
76	11508	70225	51111	38351	19444	66499	71945	05422	13442	78675	84
77	37449	30362	06694	54690	04052	53115	62757	95348	78662	11163	81
78	46515	70331	85922	38329	57015	15765	97161	17869	45349	61796	66
79	30986	81223	42416	58353	21532	30502	32305	86482	05174	07901	54
80	63798	64995	46583	09765	44160	78128	83991	42865	92520	83531	80
81	82486	84846	99254	67632	43218	50076	21361	64816	51202	88124	41
82	21885	32906	92431	09060	64297	51674	64126	62570	26123	05155	59
83	60336	98782	07408	53458	13564	59089	26445	29789	85205	41001	12
84	43937	46891	24010	25560	86355	33941	25786	54990	71899	15475	95
85	97656	63175	89303	16275	07100	92063	21942	18611	47348	20203	18
86	03299	01221	05418	38982	55758	92237	26759	86367	21216	98442	08
87	79626	06486	03574	17668	07785	76020	79924	25651	83325	88428	85
88	85636	68335	47539	03129	65651	11977	02510	26113	99447	68645	34
89	18039	14367	61337	06177	12143	46609	32989	74014	64708	00533	35
90	08362	15656	60627	36478	65648	16764	53412	09013	07832	41574	17
91	79556	29068	04142	16268	15387	12856	66227	38358	22478	73373	88
92	92608	82674	27072	32534	17075	27698	98204	63863	11951	34648	88
93	23982	25835	40055	67006	12293	02753	14827	22235	35071	99704	37
94	09915	96306	05908	97901	28395	14186	00821	80703	70426	75647	76
95	50937	33300	26695	62247	69927	76123	50842	43834	86654	70959	79
96	42488	78077	69882	61657	34136	79180	97526	43092	04098	73571	80
97	46764	86273	63003	93017	31204	36692	40202	35275	57306	55543	53
98	03237	45430	55417	63282	90816	17349	88298	90183	36600	78406	06
99	86591	81482	52667	61583	14972	90053	89534	76036	49199	43716	97
100	38534	01715	94964	87288	65680	43772	39560	12918	86537	62738	19
101	13284	16834	74151	92027	24670	36665	00770	22878	02179	51602	07
102	21224	00370	30420	03883	96648	89428	41583	17564	27395	63904	41
103	99052	47887	81085	64933	66279	80432	65793	83287	34142	13241	30
104	00199	50993	98603	38452	87890	94624	69721	57484	67501	77638	44
105	60578	06483	28733	37867	07936	98710	98539	27186	31237	80612	44
106	91240	18312	17441	01929	18163	69201	31211	54288	39296	37318	65
107	97458	14229	12063	59611	32249	90466	33216	19358	02591	54263	88
108	35249	38646	34475	72417	60514	69257	12489	51924	86871	92446	36
109	38980	46600	11759	11900	46743	27860	77940	39298	97838	95145	32
110	10750	52745	38749	87365	58959	53731	89295	59062	39404	13198	59
111	36247	27850	73958	20673	37800	63835	71051	84724	52492	22342	78
112	70994	66986	99744	72438	01174	42159	11392	20724	54322	36923	70
113	99638	94702	11463	18148	81386	80431	90628	52506	02016	85151	88
114	72055	15774	43857	99805	10419	76939	25993	03544	21560	83471	43
115	24038	65541	85788	55835	38835	59399	13790	35112	01324	39520	76
116	74976	14631	35908	28221	39470	91548	12854	30166	09073	75887	36
117	35553	71628	70189	26436	63407	91178	90348	55359	80392	41012	36
118	35676	12797	51434	82976	42010	26344	92920	92155	58807	54644	58
119	74815	67523	72985	23183	02446	63594	98924	20633	58842	85961	07
120	45246	88048	65173	50989	91060	89894	36063	32819	68559	99221	49
121	76509	47069	86378	41797	11910	49672	88575	97966	32466	10083	54
122	19689	90332	04315	21358	97248	11188	39062	63312	52496	07349	79

123	42751	35318	97513	61537	54955	08159	00337	80778	27507	95478	21
124	11946	22681	45045	13964	57517	59419	58045	44067	58716	58840	45
125	96518	48688	20996	11090	48396	57177	83867	86464	14342	21545	46

- 28 - SAMPLING GRIDS

1. A grid system should be established over the specified area (sidewalls and base). Grid point representation should be proportioned to the size of the area. It is recommended that one of the following equations be used to determine grid intervals for stationing.

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small site: GI = (A/pi)1/2

medium site: GI = (A/pi)1/2 /4

large site: GI = (Api/SF)1/2

where: A = area to be grid (sq. ft.)

GI = grid interval

SF = Site Factor, length of area to be grid (unitless)

It appears that there are logical size ranges of sites to which the three equations apply:

A) small up to 0.25 acre

B) medium: 0.25 3.0 acres, and

C) large: 3.0 acres and greater

To simplify this application, use the following chart based on an average size range of sites (1 acre = 43,560 square feet). The approximate grid ranges are provided as a quick check on numbers generated for specific sites using the above formulas.

Site Acreage*	Square Feet*	Grid Interval Ranges
up to 0.25 (small)	up to 10,890	See Part 1
0.25-3.00 (medium)	10,890-130,680	15-50 feet
3.0 and over (large)	130,680 +	30 feet plus

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* Site acreage, square footage, is total area of sidewalls and base of excavation.

2. After the grid interval is calculated, it is recommended that a scaled grid overlay be made to superimpose on the area under consideration. Some specified point (usually the southwest corner) should be designated as the 0,0 coordinate. The grid can then be adjusted to maximize sampling coverage. Some grid adjustment may be necessary for unusually shaped areas.

- 29 -STATISTICAL EVALUATION

WASTE/TREATMENT CHARACTERIZATION SAMPLINGS

Following is a step by step description of the approach used to calculate confidence limits based on the analytical data derived from the preliminary samples.

---

1. Calculate a preliminary estimate of the mean, m

m = -(X1 + X2 + X3 + ... Xn) / n

where: n = number of measurements, X = variable concentration, Xi = individual measurements

---

2. Calculate a preliminary estimate of the variance (S^2) and the standard deviation (S). Standard deviation is a function of both sampling variability and measurement variability.

S2 = [(X1-m)2+(X2-m)2+...(Xn-m)2] / n-1

---

3. Calculate the standard error of the mean (Sx). Standard error is inversely proportional to the square root of the number of samples (increasing n from 4 to 16 reduces Sx by 50%).

Sx = - S / (n)1/2

---

4. Since the concern is only whether the upper limit of a confidence interval is below or above the regulatory threshold, the lower confidence limit (LCL) need not be considered. The upper confidence limit (UCL) can be calculated using the onetailed (onesided) t values with n1 degrees of freedom derived from a table of the Student's t distribution. Where only small sized statistical samples are involved (n<30), the normal or Gaussian distribution is not accurate, and the t distribution must be used.

- 30 -

5. The 95% UCL is calculated by using the following formula and substituting the values determined above plus the appropriate t value obtained from the t table.

UCL = m + [t*0.95*(n-1)]*Sx

The term in brackets indicates a one-tailed t-test at n-1 degrees of freedom. See the t-distribution table in Attachment 2.

The UCL number resulting from this formula will indicate with a 95% probability that it is either above or below the regulatory threshold (RT) developed for the constituent being subjected to the test. If a compound does not have a specified RT, then the UCL is compared to whatever concentration is of concern (i.e., a clean up level, action level, etc). Other confidence levels can be used, based on the specific sampling situation.

If the preliminary data indicate that more samples are needed to make a hazard determination, the Lambda relationship should be used. A step by step approach to calculating the appropriate sample size follows:

1. The appropriate number of samples to be collected can be estimated by use of the Lambda relationship and then consulting a table of values and their corresponding sample size number.

Lambda = RT - m / s

The lower the calculated value, the more samples are required to maintain a certain level of confidence. Also, as approaches RT,Lambda becomes smaller, and therefore a greater sample size is

indicated for a certain level of confidence.

2. To obtain the appropriate sample size from the table of values, use the single sided value for to test at the desired significance level (for 5%, = 0.05).

3. Randomly collect any additional samples that may be needed using the same grid and random numbers sequence as the first sampling. All field and laboratory procedures should be kept as consistent as possible to lower the amount of variability in the data.

4. Use all data values to calculate new m, S, and Sx.

- 31 -

5. If the new m is equal to or greater than RT, then the contaminant is present at an unacceptable concentration and the study would be complete.

6. If m < RT and > S^2, calculate C (the criterion for determining if contamination is present at hazardous concentration). If = S^2 or m< S^2, the data must be transformed prior to calculating C.

Using the new data, C is calculated by the formula:

C = RT - m / Sx

7. Compare the calculated C value to the twotailed t value for the level of significance desired. The twotailed t-value is used because both the possibility that C is > t or that C is < t must be checked.

Use t0.95 and df (degrees of freedom) = n-1.

8. If C > t value, the contaminant is present at unacceptable concentrations and the study would be over. If C < t value, reestimate the total number of additional samples to be collected by deriving a new Lambda. Use the newly calculated values of m and S.

9. If this new number of samples is not more than 20% greater than the last set collected, there is little chance that additional samples would decrease Sx and result in the material being considered unacceptable. Therefore, the study would be complete.

- 32 - EXAMPLE

CALCULATION OF CONFIDENCE LIMITS AND LAMDA CALCULATION

Problem 1: STATISTICAL SAMPLING

A metal plating factory has been discharging process wastewaters into a large nearby swampy area for several years. This swampy area drains into a small river. The discharged wastewaters are known to be contaminated with very low levels of cadmium and chromium (i.e., the levels in the wastewater are below the facilities NPDES permit limitations). However, it has been suspected that the sediments in this swampy area may contain high levels of cadmium and chromium. Three preliminary sediment samples were taken with a Ponar dredge and analyzed to determine whether or not these sediments were contaminated with hazardous levels of these two metals. In 40 CFR 261.24, it states that a waste is hazardous under the characteristic of EP toxicity if it contains cadmium at a level 1.0 mg/L or chromium at a level 5.0 mg/L. The analysis of the three preliminary samples indicated a mean cadmium concentration of 0.37 mg/L (3 samples at 0.25, 0.51, and 0.35 mg/L) and a mean chromium

concentration of 4.66 mg/L (3 samples at 4.93, 4.21, and 4.84 mg/L). Based on this analytical data, the cadmium level is well below the regulatory threshold (RT), but the chromium level closely approaches its RT. Because large legal or monetary losses may be incurred if the sediments are declared hazardous, the analytical data must be sound and a high degree of confidence is necessary in any decision made.

QUESTIONS: Given the above scenario, answer the following questions and calculate the appropriate answers.

1. Based on the chromium data supplied

Calculate S2, S, Sx

Calculate the 95% UCL

With what degree of confidence can it be stated that the chromium concentration does not exceed the RT?

2. If more samples are deemed necessary, determine how many

Calculate the Lambda value

Calculate the appropriate number of additional samples using = 0.05 and B= 0.05

-32-

PROBLEM 1 ANSWER SHEET

Given three samples with chromium concentrations of 4.93, 4.21, and 4.84 mg/L and

m = 4.66 mg/L

(1a) Calculate S2

S2 = [(X1-m)2+(X2-m)2+...(Xn-m)2] /n-1

= [(4.93-4.66)2+(4.21-4.66)2+(4.84-4.66)2] / (3-2)

= 0.15

Calculate S

S = 0.39

Calculate Sx

Sx = S/(n)1/2 = 0.23

(1b) Calculate the 95% UCL

95% UCL = m + [t*0.95*(n-1)]*Sx

= 4.66 + (2.920)*(0.23)

= 5.33

(1c)

90% UCL = m + [t*0.90*(n-1)]*Sx

= 4.66 + (1.886)*(0.23)

= 5.09

80% UCL = m + [t*0.80*(n-1)]*Sx

= 4.66 + (1.061)*(0.23)

= 4.90

- 34 -

The preceding two calculations indicate that it can be stated with somewhere between 80% and 90% confidence that the chromium concentration does not exceed the RT. This degree of confidence may not be sufficient to meet the needs of the sampling plan. Therefore, more samples may need to be taken.

2a. Calculate the Lambda value

Lambda = (RT - m) / S

= (5.0 - 4.66) / 0.39 = 0.87

2b. Calculate the number of additional samples

Using Attachment 2, *Number of Observations for t Test of Mean*, page 36 of this Guidance Document, using a single-sided test with =0.05 and =0.05, approximately 15 to 17 total samples need to be collected. Therefore, based on the three preliminary samples that were collected, an additional 13 samples need to be taken.

- 35 -

Cumulative t Distribution

one-tailed	0.550	0.750	0.080	0.900	0.950	0.975	0.990
two-tailed	0.100	0.500	0.600	0.800	0.900	0.950	0.980
1	0.158	1.000	1.376	3.078	6.314	12.706	31.821
2	0.142	0.816	1.061	1.886	2.920	4.303	6.925
3	0.137	0.765	0.978	1.638	2.353	3.182	4.541
4	0.134	0.741	0.941	1.533	2.132	2.776	3.747
5	0.132	0.727	0.920	1.476	2.015	2.571	3.365
6	0.131	0.718	0.906	1.440	1.943	2.447	3.143
7	0.130	0.711	0.896	1.415	1.895	2.365	2.998
8	0.130	0.706	0.889	1.397	1.860	2.306	2.896
9	0.129	0.703	0.883	1.383	1.833	2.262	2.821
10	0.129	0.700	0.879	1.372	1.812	2.228	2.764
11	0.129	0.697	0.876	1.363	1.796	2.201	2.718

12	0.128	0.695	0.873	1.356	1.782	2.179	2.681
13	0.128	0.694	0.870	1.350	1.771	2.160	2.650
14	0.128	0.692	0.868	1.345	1.761	2.145	2.624
15	0.128	0.691	0.866	1.341	1.753	2.131	2.602
16	0.128	0.690	0.865	1.337	1.746	2.120	2.583
17	0.128	0.689	0.863	1.333	1.740	2.110	2.567
18	0.127	0.688	0.862	1.330	1.734	2.101	2.552
19	0.127	0.688	0.861	1.328	1.729	2.093	2.539
20	0.127	0.687	0.860	1.325	1.725	2.086	2.528
21	0.127	0.686	0.859	1.323	1.721	2.080	2.518
22	0.127	0.686	0.858	1.321	1.717	2.074	2.508
23	0.127	0.685	0.858	1.319	1.714	2.069	2.500
24	0.127	0.685	0.857	1.318	1.711	2.064	2.492
25	0.127	0.684	0.856	1.316	1.708	2.060	2.485
26	0.127	0.684	0.856	1.315	1.706	2.056	2.479
27	0.127	0.684	0.855	1.314	1.703	2.052	2.473
28	0.127	0.683	0.855	1.313	1.701	2.048	2.467
29	0.127	0.683	0.854	1.311	1.699	2.045	2.462
30	0.127	0.683	0.854	1.310	1.697	2.042	2.457
40	0.126	0.681	0.851	1.303	1.684	2.021	2.423
60	0.126	0.679	0.848	1.296	1.671	2.000	2.390
120	0.126	0.677	0.845	1.289	1.658	1.980	2.358
	0.126	0.674	0.842	1.282	1.645	1.960	2.326

NOTE: For one-tailed distributions a/2 = 1-p
For two-tailed distributions a = 1-p

- 36 -

NUMBER OF OBSERVATIONS FOR t TEST OF MEAN

Level for t Test															A
	Double-sided A=0.01					A=0.02					Single-sided A=0.005				
											A=0.05				
B=0.01	0.05	0.1	0.2	0.5		0.01	0.05	0.1	0.2	0.5		0.01	0.05	0.1	0.2
LAMBDA															
0.15															
0.20										139					
0.25				110						90				128	
0.30			134	78					115	63			119	90	
0.35		125	99	58				109	85	47		109	88	67	
0.40	115	97	77	45			101	85	66	37	117	84	68	51	
0.45	92	77	62	37		110	81	68	53	30	93	67	54	41	
0.50	100 75	63	51	30		90	66	55	43	25	76	54	44	34	
0.55	83 63	53	42	26		75	55	46	36	21	63	45	37	28	
0.60	71 53	45	36	22		63	47	39	31	18	53	38	32	24	
0.65	61 46	39	31	20		55	41	34	27	16	46	33	27	21	
0.70	53 40	34	28	17		47	35	30	24	14	40	29	24	19	
0.75	47 36	30	25	16		42	31	27	21	13	35	26	21	16	
0.80	41 32	27	22	14		37	28	24	19	12	31	22	19	15	
0.85	37 29	24	20	13		33	25	21	17	11	28	21	17	13	
0.90	34 26	22	18	12		29	23	19	16	10	25	19	16	12	
0.95	31 24	20	17	11		27	21	18	14	9	23	17	14	11	
1.00	28 22	19	16	10		25	19	16	13	9	21	16	13	10	
1.1	24 19	16	14	9		21	16	14	12	8	18	13	11	9	
1.2	21 16	14	12	8		18	14	12	10	7	15	12	10	8	
1.3	18 15	13	11	8		16	13	11	9	6	14	10	9	7	
1.4	16 13	12	10	8		14	11	10	9	6	12	9	8	7	
1.5	15 12	11	9	7		13	10	9	8	6	11	8	7	6	
1.6	13 11	10	8	6		12	10	9	7	5	10	8	7	6	
1.7	12 10	9	8	6		11	9	8	7		9	7	6	5	
1.8	12 10	9	8	6		10	8	7	7		8	7	6		

1.9	11	9	8	7	6	10	8	7	6	8	6	6
2.0	10	8	8	7	5	9	7	7	6	7	6	5
2.1	10	8	7	7		8	7	6	6	7	6	
2.2	9	8	7	6		8	7	6	5	7	6	
2.3	9	7	7	6		8	6	6		6	5	
2.4	8	7	7	6		7	6	6		6		
2.5	8	7	6	6		7	6	6		6		
3.0	7	6	6	5		6	5	5		5		
3.5	6	5	5			5						
4.0	6											

99% confidence

SAMPLING PROCESS STREAMS

Although sampling is generally thought to occur on a pile of material or over an area of treated soil, other schemes are possible. The most common instance is when the material is to be sampled at the point of generation. This is the preferred method, since it is most representative of the material under study. The lack of exposure to elements that might cause chemical degradation and/or leaching will result in material most indicative of actual conditions.

A sampling point along the material conveyor that can be fairly easily and safely reached should be chosen. It should be in an area where the entire belt can be accessed for sampling. Under this scenario, a temporal, rather than a spatial, approach needs to be used.

Time stratum should be established over the course of the process day. Ideally, the entire active time of the line should be included in the sampling scheme. Once time strata are chosen, the random numbers table can be used to establish sampling times. For a four hour period, a point somewhere on the table would be chosen and every number greater than 0 but less than 240 would be selected until the number of samples for that strata were obtained. The number would relate to time in minutes. This would be added to the starting time for that strata to determine the time of sampling.

If the time strata chosen are of unequal lengths, the number of samples chosen from any one strata should reflect the percentage contribution that strata makes to the time frame as a whole. For example, if for a 24 hour operating time, strata 1 is 4 hours and strata 2 is 8 hours, strata 2 should have twice as many samples as strata 1.

When the appropriate sampling time arrives, the material from the conveyor belt point that had been identified would be removed. This material should be well mixed and a subsample taken for inclusion in the jar for lab analysis. An example of the use of this protocol is attached.

- 38 - RANDOM TIME WASTE SAMPLING EXAMPLE

	Sampling Point	Random Minute	Time
Stratum #1 6:00 to 8:00 hours	1	28	6:28
	2	62	7:02
	3	99	7:39
	4	112	7:52
Stratum #2 8:00 to 20:00 hours	1	11	8:11
	2	107	9:47
	3	156	10:36
	4	173	10:53

	5	296	12:56
	6	313	13:13
	7	398	14:38
	8	497	16:17
	9	555	17:15
	10	600	18:00
	11	637	18:37
	12	706	19:46
Stratum #3 20:00 to 22:00 hours	1	13	20:13
	2	52	20:52
	3	88	21:28
	4	108	21:48
Stratum #4 8:00 to 20:00 hours	1	48	22:48
	2	113	23:53
	3	153	24:33
	4	189	1:09
	5	227	1:47
	6	290	2:49
	7	314	3:14
	8	474	5:44

- 39 - A T T A C H M E N T 3

TOLERANCE FACTORS (K)

TOLERANCE FACTORS (K) FOR ONE-SIDED NORMAL TOLERANCE INTERVALS WITH PROBABILITY LE
Y = 0.95 AND COVERAGE P = 95%

n	K	n	K
3	7.655	75	1.972
4	5.145	100	1.924
5	4.202	125	1.891
6	3.707	150	1.868
7	3.399	175	1.850
8	3.188	200	1.836
9	3.031	225	1.824
10	2.911	250	1.814
11	2.815	275	1.806
12	2.736	300	1.799
13	2.670	325	1.792
14	2.614	350	1.787
15	2.566	375	1.782
16	2.523	400	1.777
17	2.486	425	1.773
18	2.453	450	1.769
19	2.423	475	1.766
20	2.396	500	1.763
21	2.371	525	1.760
22	2.350	550	1.757
23	2.329	575	1.754
24	2.309	600	1.752
25	2.292	625	1.750
30	2.220	650	1.748
35	2.166	675	1.746
40	2.126	700	1.744
45	2.092	725	1.742
50	2.065	750	1.740
55	2.036	775	1.739
60	2.017	800	1.737

65	2.000	825	0.736
70	1.986	850	1.734
		875	1.733
		900	1.732
		925	1.731
		950	1.729
		975	1.728
		1,000	1.727

SOURCE: FOR SAMPLE SIZES ≤ 50: Lieberman, Gerald F. 1958. "Tables for One-sided Statistical Tolerance Limits." *Industrial Quality Control.* Vol. XIV, No. 10.

FOR SAMPLE SIZES ≥ 50: K values were calculated from large sample approximation.

NTIS Document PB-89-151-047

- 40 - A T T A C H M E N T 4

WASTE MANAGEMENT DIVISION'S CLEAN CLOSURE CERTIFICATION CHECKLIST

This checklist was developed to review RCRA clean closures. Due to direct reference to 40 CFR, Part 264, Subpart G, by Act 64, Rule 613; Act 64 closures should also be evaluated by this checklist.

Documentation supporting the owners/operators and the independent registered professional engineer's certification can be requested under 40 CFR, 264.115 and 265.115 (as of October 29, 1986). The owner/operator must submit at least four copies of certification documentation.

The checklist identifies items recommended to properly evaluate a closure certification. These items are not "absolutes." Other information or substitutions may be provided which technically justify and certify a "clean closure."

This checklist can be used for land disposal, storage, and treatment facilities. Several of the items would not be required for storage and/or treatment facilities where testing was minimal. Items 1 through 5 would be required for all closures. Items 6 through 11 would be optional for storage and/or treatment facilities, dependent on extent of testing required. Land disposal facilities would require all items listed.

1. Manifests (or some type of manifest/waste removal summary) of where and how much waste was shipped.

2. Certification statement is needed by the owner/operator AND an independent registered engineer. All independent registered professional engineer certificates must have an original stamp on at least one copy.

3. Summary of decontamination procedures (pressure wash, Steam clean, etc.) and how the resultant waste water was disposed.

4. Summary analysis (include conditions of haul roads, time table, soil and groundwater results, weather conditions, runoff controls, equipment decontamination, etc.).

5. Results of all tests used to determine clean closure (charts, tables, lab sheets).

6. Statistical comparisons on sampling results compared to cleanup criteria (this should include full

computations on background and statistical analysis).

7. Sampling and analysis procedures (specify references).

8. Final depth and elevations of excavations of wastes and soils.

- 41 -

9. Properly labeled and easily identified sampling locations and grid stations (map) including background stations.

10. Groundwater data (and statistical evaluation) used to determine if groundwater degradation has occurred (usually four sets of replicate analysis compared to sampling event after closure activities). Monitor well construction details and sampling and analysis procedures may be required if documentation is not in the file.

11. Summary of final restoration of excavated area...

information on fill material used and/or future land use outline. If clean closure cannot be achieved (e.g., contaminated soils to water table and groundwater results show contamination). This summary item should be used to address the need for any post closure program and/or corrective action.

12. A copy of all field notes pertaining to these closure activities.

13. A copy of the approved closure plan and letter of closure plan approval.

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top of page

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ATTACHMENT B

APPENDIX IX CONSTITUENT LIST

APPENDIX IX TO PART 264—GROUND-WATER MONITORING LIST[1]

GROUND-WATER MONITORING LIST[1]

Common name[2]	CAS RN[3]	Chemical abstracts service index name[4]	Suggested methods[5]	PQL (µg/L)[6]
Acenaphthene	83–32–9	Acenaphthylene, 1,2-dihydro-	8100	200
			8270	10
Acenaphthylene	208–96–8	Acenaphthylene	8100	200
			8270	10
Acetone	67–64–1	2-Propanone	8240	100
Acetophenone	98–86–2	Ethanone, 1-phenyl-	8270	10
Acetonitrile; Methyl cyanide	75–05–8	Acetonitrile	8015	100
2-Acetylaminofluorene; 2-AAF	53–96–3	Acetamide, N-9H-fluoren-2-yl-	8270	10
Acrolein	107–02–8	2-Propenal	8030	5
			8240	5
Acrylonitrile	107–13–1	2-Propenenitrile	8030	5
			8240	5
Aldrin	309–00–2	1,4:5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexachloro- 1,4,4a,5,8,8a-hexahydro- (1α,4α, 4aβ, 5α,8α,8aβ)-	8080	0.05
			8270	10
Allyl chloride	107–05–1	1-Propene, 3-chloro-	8010	5
			8240	100
4-Aminobiphenyl	92–67–1	[1,1'-Biphenyl]- 4-amine	8270	10
Aniline	62–53–3	Benzenamine	8270	10
Anthracene	120–12–7	Anthracene	8100	200
			8270	10
Antimony	(Total)	Antimony	6010	300
			7040	2,000
			7041	30
Aramite	140–57–8	Sulfurous acid, 2-chloroethyl 2-[4-(1,1-dimethylethyl)phenoxy]-1-methylethyl ester.	8270	10
Arsenic	(Total)	Arsenic	6010	500
			7060	10
			7061	20
Barium	(Total)	Barium	6010	20
			7080	1,000
Benzene	71–43–2	Benzene	8020	2
			8240	5
Benzo[a]anthracene; Benzanthracene.	56–55–3	Benz[a]anthracene	8100	200
			8270	10
Benzo[b]fluoranthene	205–99–2	Benz[e]acephenanthrylene	8100	200
			8270	10
Benzo[k]fluoranthene	207–08–9	Benzo[k]fluoranthene	8100	200
			8270	10
Benzo[ghi]perylene	191–24–2	Benzo[ghi]perylene	8100	200
			8270	10
Benzo[a]pyrene	50–32–8	Benzo[a]pyrene	8100	200
			8270	10
Benzyl alcohol	100–51–6	Benzenemethanol	8270	20
Beryllium	(Total)	Beryllium	6010	3
			7090	50
			7091	2
alpha-BHC	319–84–6	Cyclohexane, 1,2,3,4,5,6-hexachloro-,(1α, 2α,3β, 4α,5β,6β)-	8080	0.05
			8250	10
beta-BHC	319–85–7	Cyclohexane, 1,2,3,4,5,6-hexachloro-,(1α,2β, 3α,4β, 5α,6β)-	8080	0.05
			8250	40
delta-BHC	319–86–8	Cyclohexane, 1,2,3,4,5,6-hexachloro-,(1α,2α, 3α, 4β,5α,6β)-	8080	0.1
			8250	30
gamma-BHC; Lindane	58–89–9	Cyclohexane, 1,2,3,4,5,6-hexachloro-,(1α, 2α, 3β, 4α,5α,6β)-	8080	0.05
			8250	10
Bis(2-chloroethoxy)methane	111–91–1	Ethane, 1,1'-[methylenebis (oxy)]bis [2-chloro-	8270	10
Bis(2-chloroethyl)ether	111–44–4	Ethane, 1,1'-oxybis[2-chloro-	8270	10
Bis(2-chloro-1-methylethyl) ether; 2,2'-Dichlorodiisopropyl ether.	108–60–1	Propane, 2,2'-oxybis[1-chloro-	8010	100
			8270	10
Bis(2-ethylhexyl) phthalate	117–81–7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl)ester	8060	20
			8270	10
Bromodichloromethane	75–27–4	Methane, bromodichloro-	8010	1
			8240	5

GROUND-WATER MONITORING LIST [1]—Continued

Common name [2]	CAS RN [3]	Chemical abstracts service index name [4]	Suggested methods [5]	PQL (µg/L) [6]
Bromoform; Tribromomethane	75-25-2	Methane, tribromo-	8010	2
			8240	5
4-Bromophenyl phenyl ether	101-55-3	Benzene, 1-bromo-4-phenoxy-	8270	10
Butyl benzyl phthalate; Benzyl butyl phthalate	85-68-7	1,2-Benzenedicarboxylic acid, butyl phenylmethyl ester.	8060	5
			8270	10
Cadmium	(Total)	Cadmium	6010	40
			7130	50
			7131	1
Carbon disulfide	75-15-0	Carbon disulfide	8240	5
Carbon tetrachloride	56-23-5	Methane, tetrachloro-	8010	1
			8240	5
Chlordane	57-74-9	4,7-Methano-1H-indene, 1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a- hexahydro-.	8080	0.1
			8250	10
p-Chloroaniline	106-47-8	Benzenamine, 4-chloro-	8270	20
Chlorobenzene	108-90-7	Benzene, chloro-	8010	2
			8020	2
			8240	5
Chlorobenzilate	510-15-6	Benzeneacetic acid, 4-chloro-α-(4-chlorophenyl)-α-hydroxy-, ethyl ester.	8270	10
p-Chloro-m-cresol	59-50-7	Phenol, 4-chloro-3-methyl-	8040	5
			8270	20
Chloroethane; Ethyl chloride	75-00-3	Ethane, chloro-	8010	5
			8240	10
Chloroform	67-66-3	Methane, trichloro-	8010	0.5
			8240	5
2-Chloronaphthalene	91-58-7	Naphthalene, 2-chloro-	8120	10
			8270	10
2-Chlorophenol	95-57-8	Phenol, 2-chloro-	8040	5
			8270	10
4-Chlorophenyl phenyl ether	7005-72-3	Benzene, 1-chloro-4-phenoxy-	8270	10
Chloroprene	126-99-8	1,3-Butadiene, 2-chloro-	8010	50
			8240	5
Chromium	(Total)	Chromium	6010	70
			7190	500
			7191	10
Chrysene	218-01-9	Chrysene	8100	200
			8270	10
Cobalt	(Total)	Cobalt	6010	70
			7200	500
			7201	10
Copper	(Total)	Copper	6010	60
			7210	200
m-Cresol	108-39-4	Phenol, 3-methyl-	8270	10
o-Cresol	95-48-7	Phenol, 2-methyl-	8270	10
p-Cresol	106-44-5	Phenol, 4-methyl-	8270	10
Cyanide	57-12-5	Cyanide	9010	40
2,4-D; 2,4-Dichlorophenoxyacetic acid.	94-75-7	Acetic acid, (2,4-dichlorophenoxy)-	8150	10
4,4'-DDD	72-54-8	Benzene 1,1'-(2,2-dichloroethylidene) bis[4-chloro-	8080	0.1
			8270	10
4,4'-DDE	72-55-9	Benzene, 1,1'-(dichloroethenylidene) bis[4-chloro-	8080	0.05
			8270	10
4,4'-DDT	50-29-3	Benzene, 1,1'-(2,2,2-trichloroethylidene) bis[4-chloro-	8080	0.1
			8270	10
Diallate	2303-16-4	Carbamothioic acid, bis(1-methylethyl)- , S- (2,3-dichloro-2-propenyl) ester.	8270	10
Dibenz[a,h]anthracene	53-70-3	Dibenz[a,h]anthracene	8100	200
			8270	10
Dibenzofuran	132-64-9	Dibenzofuran	8270	10
Dibromochloromethane; Chlorodibromomethane	124-48-1	Methane, dibromochloro-	8010	1
			8240	5
1,2-Dibromo-3-chloropropane; DBCP.	96-12-8	Propane, 1,2-dibromo-3-chloro-	8010	100
			8240	5
			8270	10
1,2-Dibromoethane; Ethylene dibromide.	106-93-4	Ethane, 1,2-dibromo-	8010	10
			8240	5
Di-n-butyl phthalate	84-74-2	1,2-Benzenedicarboxylic acid, dibutyl ester	8060	5
			8270	10
o-Dichlorobenzene	95-50-1	Benzene, 1,2-dichloro-	8010	2
			8020	5

GROUND-WATER MONITORING LIST [1]—Continued

Common name [2]	CAS RN [3]	Chemical abstracts service index name [4]	Suggested methods [5]	PQL (µg/L) [6]
			8120	10
			8270	10
m-Dichlorobenzene	541-73-1	Benzene, 1,3-dichloro-	8010	5
			8020	5
			8120	10
			8270	10
p-Dichlorobenzene	106-46-7	Benzene, 1,4-dichloro-	8010	2
			8020	5
			8120	15
			8270	10
3,3'-Dichlorobenzidine	91-94-1	[1,1'-Biphenyl]- 4,4'- diamine, 3,3'-dichloro-	8270	20
trans-1,4-Dichloro-2-butene	110-57-6	2-Butene, 1,4-dichloro-, (E)-	8240	5
Dichlorodifluoromethane	75-71-8	Methane, dichlorodifluoro-	8010	10
			8240	5
1,1-Dichloroethane	75-34-3	Ethane, 1,1-dichloro-	8010	1
			8240	5
1,2-Dichloroethane; Ethylene dichloride.	107-06-2	Ethane, 1,2-dichloro-	8010	0.5
			8240	5
1,1-Dichloroethylene; Vinylidene chloride.	75-35-4	Ethene, 1,1-dichloro-	8010	1
			8240	5
trans-1,2-Dichloroethylene	156-60-5	Ethene, 1,2-dichloro-, (E)-	8010	1
			8240	5
2,4-Dichlorophenol	120-83-2	Phenol, 2,4-dichloro-	8040	5
			8270	10
2,6-Dichlorophenol	87-65-0	Phenol, 2,6-dichloro-	8270	10
1,2-Dichloropropane	78-87-5	Propane, 1,2-dichloro-	8010	0.5
			8240	5
cis-1,3-Dichloropropene	10061-01-5	1-Propene, 1,3-dichloro-, (Z)-	8010	20
			8240	5
trans-1,3-Dichloropropene	10061-02-6	1-Propene, 1,3-dichloro-, (E)-	8010	5
			8240	5
Dieldrin	60-57-1	2,7:3,6-Dimethanonaphth [2,3-b]oxirene, 3,4,5,6,9,9-hexachloro- 1a,2,2a,3,6,6a,7,7a- octahydro-, (1aα,2β, 2aα, 3β,6β,6aα,7β,7aα)-	8080	0.05
			8270	10
Diethyl phthalate	84-66-2	1,2-Benzenedicarboxylic acid, diethyl ester	8060	5
			8270	10
O,O-Diethyl O-2-pyrazinyl phosphorothioate; Thionazin	297-97-2	Phosphorothioic acid, O,O-diethyl O-pyrazinyl ester	8270	10
Dimethoate	60-51-5	Phosphorodithioic acid, O,O-dimethyl S-[2-(methylamino)-2-oxoethyl] ester.	8270	10
p-(Dimethylamino)azobenzene	60-11-7	Benzenamine, N,N-dimethyl-4-(phenylazo)-	8270	10
7,12-Dimethylbenz[a]anthracene	57-97-6	Benz[a]anthracene, 7,12-dimethyl-	8270	10
3,3'-Dimethylbenzidine	119-93-7	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethyl-	8270	10
alpha, alpha-Dimethylphenethylamine.	122-09-8	Benzeneethanamine, α,α-dimethyl-	8270	10
2,4-Dimethylphenol	105-67-9	Phenol, 2,4-dimethyl-	8040	5
			8270	10
Dimethyl phthalate	131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester	8060	5
			8270	10
m-Dinitrobenzene	99-65-0	Benzene, 1,3-dinitro-	8270	10
4,6-Dinitro-o-cresol	534-52-1	Phenol, 2-methyl-4,6-dinitro-	8040	150
			8270	50
2,4-Dinitrophenol	51-28-5	Phenol, 2,4-dinitro-	8040	150
			8270	50
2,4-Dinitrotoluene	121-14-2	Benzene, 1-methyl-2,4-dinitro-	8090	0.2
			8270	10
2,6-Dinitrotoluene	606-20-2	Benzene, 2-methyl-1,3-dinitro-	8090	0.1
			8270	10
Dinoseb; DNBP; 2-sec-Butyl-4,6-dinitrophenol	88-85-7	Phenol, 2-(1-methylpropyl)-4,6-dinitro-	8150	1
			8270	10
Di-n-octyl phthalate	117-84-0	1,2-Benzenedicarboxylic acid, dioctyl ester	8060	30
			8270	10
1,4-Dioxane	123-91-1	1,4-Dioxane	8015	150
Diphenylamine	122-39-4	Benzenamine, N-phenyl-	8270	10
Disulfoton	298-04-4	Phosphorodithioic acid, O,O-diethyl S-[2-(ethylthio)ethyl]ester	8140	2
			8270	10
Endosulfan I	959-98-8	6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-, 3-oxide, (3α,5aβ,6α,9α,9aβ)-.	8080	0.1
			8250	10

GROUND-WATER MONITORING LIST [1]—Continued

Common name [2]	CAS RN [3]	Chemical abstracts service index name [4]	Suggested methods [5]	PQL (µg/L) [6]
Endosulfan II	33213–65–9	6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro- 1,5,5a,6,9,9a-hexahydro-, 3-oxide, (3α,5aα, 6β,9β, 9aα)-	8080	0.05
Endosulfan sulfate	1031–07–8	6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro- 1,5,5a,6,9,9a-hexahydro-, 3,3-dioxide.	8080 8270	0.5 10
Endrin	72–20–8	2,7:3,6-Dimethanonaphth[2,3-b]oxirene, 3,4,5,6,9,9-hexachloro- 1a,2,2a,3,6,6a,7,7a- octahydro-, (1aα, 2β,2aβ, 3α,6α, 6aβ,7β, 7aα)-	8080 8250	0.1 10
Endrin aldehyde	7421–93–4	1,2,4-Methenocyclopenta[cd]pentalene- 5-carboxaldehyde, 2,2a,3,3,4,7-hexachlorodecahydro-, (1α,2β, 2aβ,4β, 4aβ,5β,6aβ,6bβ,7R*)-	8080 8270	0.2 10
Ethylbenzene	100–41–4	Benzene, ethyl-	8020 8240	2 5
Ethyl methacrylate	97–63–2	2-Propenoic acid, 2-methyl-, ethyl ester	8015 8240 8270	10 5 10
Ethyl methanesulfonate	62–50–0	Methanesulfonic acid, ethyl ester	8270	10
Famphur	52–85–7	Phosphorothioic acid, O-[4-[(dimethylamino)sulfonyl]phenyl]-O,O-dimethyl ester.	8270	10
Fluoranthene	206–44–0	Fluoranthene	8100 8270	200 10
Fluorene	86–73–7	9H-Fluorene	8100 8270	200 10
Heptachlor	76–44–8	4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-	8080 8270	0.05 10
Heptachlor epoxide	1024–57–3	2,5-Methano-2H-indeno[1,2-b]oxirene, 2,3,4,5,6,7,7-heptachloro-1a,1b,5,5a,6,6a,-hexahydro-, (1aα,1bβ,2α,5α,5aβ,6β,6aα)	8080 8270	1 10
Hexachlorobenzene	118–74–1	Benzene, hexachloro-	8120 8270	0.5 10
Hexachlorobutadiene	87–68–3	1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	8120 8270	5 10
Hexachlorocyclopentadiene	77–47–4	1,3-Cyclopentadiene, 1,2,3,4,5,5-hexachloro-	8120 8270	5 10
Hexachloroethane	67–72–1	Ethane, hexachloro-	8120 8270	0.5 10
Hexachlorophene	70–30–4	Phenol, 2,2'-methylenebis[3,4,6-trichloro-	8270	10
Hexachloropropene	1888–71–7	1–Propene, 1,1,2,3,3,3-hexachloro-	8270	10
2-Hexanone	591–78–6	2-Hexanone	8240	50
Indeno(1,2,3-cd)pyrene	193–39–5	Indeno[1,2,3-cd]pyrene	8100 8270	200 10
Isobutyl alcohol	78–83–1	1-Propanol, 2-methyl-	8015	50
Isodrin	465–73–6	1,4,5,8-Dimethanonaphthalene,1,2,3,4,10,10-hexachloro- 1,4,4a,5,8,8a hexahydro-(1α, 4α, 4aβ, 5β, 8β,8aβ)-.	8270	10
Isophorone	78–59–1	2-Cyclohexen-1-one, 3,5,5-trimethyl-	8090 8270	60 10
Isosafrole	120–58–1	1,3-Benzodioxole, 5-(1-propenyl)-	8270	10
Kepone	143–50–0	1,3,4-Metheno-2H-cyclobuta- [cd]pentalen-2-one, 1,1a,3,3a,4,5,5,5a,5b,6- decachlorooctahydro-	8270	10
Lead	(Total)	Lead	6010 7420 7421	40 1,000 10
Mercury	(Total)	Mercury	7470	2
Methacrylonitrile	126–98–7	2-Propenenitrile, 2-methyl-	8015 8240	5 5
Methapyrilene	91–80–5	1,2,Ethanediamine, N,N-dimethyl-N'-2- pyridinyl- N'-(2-thienylmethyl)-.	8270	10
Methoxychlor	72–43–5	Benzene, 1,1'-(2,2,2,trichloroethylidene)bis[4-methoxy-.	8080 8270	2 10
Methyl bromide; Bromomethane	74–83–9	Methane, bromo-	8010 8240	20 10
Methyl chloride; Chloromethane	74–87–3	Methane, chloro-	8010 8240	1 10
3-Methylcholanthrene	56–49–5	Benz[j]aceanthrylene, 1,2-dihydro-3-methyl-	8270	10
Methylene bromide; Dibromomethane.	74–95–3	Methane, dibromo-	8010 8240	15 5

GROUND-WATER MONITORING LIST [1]—Continued

Common name [2]	CAS RN [3]	Chemical abstracts service index name [4]	Suggested methods [5]	PQL (µg/L) [6]
Methylene chloride; Dichloromethane.	75–09–2	Methane, dichloro-	8010 8240	5 5
Methyl ethyl ketone; MEK	78–93–3	2-Butanone	8015 8240	10 100
Methyl iodide; Iodomethane	74–88–4	Methane, iodo-	8010 8240	40 5
Methyl methacrylate	80–62–6	2-Propenoic acid, 2-methyl-, methyl ester	8015 8240	2 5
Methyl methanesulfonate	66–27–3	Methanesulfonic acid, methyl ester	8270	10
2-Methylnaphthalene	91–57–6	Naphthalene, 2-methyl-	8270	10
Methyl parathion; Parathion methyl.	298–00–0	Phosphorothioic acid, O,O-dimethyl O-(4-nitrophenyl) ester.	8140 8270	0.5 10
4-Methyl-2-pentanone; Methyl isobutyl ketone.	108–10–1	2-Pentanone, 4-methyl-	8015 8240	5 50
Naphthalene	91–20–3	Naphthalene	8100 8270	200 10
1,4-Naphthoquinone	130–15–4	1,4-Naphthalenedione	8270	10
1-Naphthylamine	134–32–7	1-Naphthalenamine	8270	10
2-Naphthylamine	91–59–8	2-Naphthalenamine	8270	10
Nickel	(Total)	Nickel	6010 7520	50 400
o-Nitroaniline	88–74–4	Benzenamine, 2-nitro-	8270	50
m-Nitroaniline	99–09–2	Benzenamine, 3-nitro-	8270	50
p-Nitroaniline	100–01–6	Benzenamine, 4-nitro-	8270	50
Nitrobenzene	98–95–3	Benzene, nitro-	8090 8270	40 10
o-Nitrophenol	88–75–5	Phenol, 2-nitro-	8040 8270	5 10
p-Nitrophenol	100–02–7	Phenol, 4-nitro-	8040 8270	10 50
4-Nitroquinoline 1-oxide	56–57–5	Quinoline, 4-nitro-, 1-oxide	8270	10
N-Nitrosodi-n-butylamine	924–16–3	1-Butanamine, N-butyl-N-nitroso-	8270	10
N-Nitrosodiethylamine	55–18–5	Ethanamine, N-ethyl-N-nitroso-	8270	10
N-Nitrosodimethylamine	62–75–9	Methanamine, N-methyl-N-nitroso-	8270	10
N-Nitrosodiphenylamine	86–30–6	Benzenamine, N-nitroso-N-phenyl-	8270	10
N-Nitrosodipropylamine; Di-n-propylnitrosamine.	621–64–7	1-Propanamine, N-nitroso-N-propyl-	8270	10
N-Nitrosomethylethylamine	10595–95–6	Ethanamine, N-methyl-N-nitroso-	8270	10
N-Nitrosomorpholine	59–89–2	Morpholine, 4-nitroso-	8270	10
N-Nitrosopiperidine	100–75–4	Piperidine, 1-nitroso-	8270	10
N-Nitrosopyrrolidine	930–55–2	Pyrrolidine, 1-nitroso-	8270	10
5-Nitro-o-toluidine	99–55–8	Benzenamine, 2-methyl-5-nitro-	8270	10
Parathion	56–38–2	Phosphorothioic acid, O,O-diethyl-O-(4-nitrophenyl) ester	8270	10
Polychlorinated biphenyls; PCBs	See Note 7	1,1'-Biphenyl, chloro derivatives	8080 8250	50 100
Polychlorinated dibenzo-p-dioxins; PCDDs.	See Note 8	Dibenzo[b,e][1,4]dioxin, chloro derivatives	8280	0.01
Polychlorinated dibenzofurans; PCDFs.	See Note 9	Dibenzofuran, chloro derivatives	8280	0.01
Pentachlorobenzene	608–93–5	Benzene, pentachloro-	8270	10
Pentachloroethane	76–01–7	Ethane, pentachloro-	8240 8270	5 10
Pentachloronitrobenzene	82–68–8	Benzene, pentachloronitro-	8270	10
Pentachlorophenol	87–86–5	Phenol, pentachloro-	8040 8270	5 50
Phenacetin	62–44–2	Acetamide, N-(4-ethoxyphenyl)	8270	10
Phenanthrene	85–01–8	Phenanthrene	8100 8270	200 10
Phenol	108–95–2	Phenol	8040 8270	1 10
p-Phenylenediamine	106–50–3	1,4-Benzenediamine	8270	10
Phorate	298–02–2	Phosphorodithioic acid, O,O-diethyl S-[(ethylthio)methyl] ester	8140 8270	2 10
2-Picoline	109–06–8	Pyridine, 2-methyl-	8240 8270	5 10
Pronamide	23950–58–5	Benzamide, 3,5-dichloro-N-(1,1-dimethyl-2-propynyl)-	8270	10
Propionitrile; Ethyl cyanide	107–12–0	Propanenitrile	8015 8240	60 5
Pyrene	129–00–0	Pyrene	8100	200

GROUND-WATER MONITORING LIST [1]—Continued

Common name [2]	CAS RN [3]	Chemical abstracts service index name [4]	Suggested methods [5]	PQL (µg/L) [6]
Pyridine	110-86-1	Pyridine	8270	10
			8240	5
			8270	10
Safrole	94-59-7	1,3-Benzodioxole, 5-(2-propenyl)-	8270	10
Selenium	(Total)	Selenium	6010	750
			7740	20
			7741	20
Silver	(Total)	Silver	6010	70
			7760	100
Silvex; 2,4,5-TP	93-72-1	Propanoic acid, 2-(2,4,5-trichlorophenoxy)-	8150	2
Styrene	100-42-5	Benzene, ethenyl-	8020	1
			8240	5
Sulfide	18496-25-8	Sulfide	9030	10,000
2,4,5-T; 2,4,5-Trichlorophenoxyacetic acid.	93-76-5	Acetic acid, (2,4,5- trichlorophenoxy)-	8150	2
2,3,7,8-TCDD; 2,3,7,8-Tetrachlorodibenzo-p- dioxin	1746-01-6	Dibenzo[b,e][1,4]dioxin, 2,3,7,8-tetrachloro-	8280	0.005
1,2,4,5-Tetrachlorobenzene	95-94-3	Benzene, 1,2,4,5-tetrachloro-	8270	10
1,1,1,2-Tetrachloroethane	630-20-6	Ethane, 1,1,1,2-tetrachloro-	8010	5
			8240	5
1,1,2,2-Tetrachloroethane	79-34-5	Ethane, 1,1,2,2-tetrachloro-	8010	0.5
			8240	5
Tetrachloroethylene; Perchloroethylene; Tetrachloroethene.	127-18-4	Ethene, tetrachloro-	8010	0.5
			8240	5
2,3,4,6-Tetrachlorophenol	58-90-2	Phenol, 2,3,4,6-tetrachloro-	8270	10
Tetraethyl dithiopyrophosphate; Sulfotepp.	3689-24-5	Thiodiphosphoric acid ($[(HO)_2 P(S)]_2 O$), tetraethyl ester	8270	10
Thallium	(Total)	Thallium	6010	400
			7840	1,000
			7841	10
Tin	(Total)	Tin	7870	8,000
Toluene	108-88-3	Benzene, methyl-	8020	2
			8240	5
o-Toluidine	95-53-4	Benzenamine, 2-methyl-	8270	10
Toxaphene	8001-35-2	Toxaphene	8080	2
			8250	10
1,2,4-Trichlorobenzene	120-82-1	Benzene, 1,2,4-trichloro-	8270	10
1,1,1-Trichloroethane; Methylchloroform.	71-55-6	Ethane, 1,1,1-trichloro-	8240	5
1,1,2-Trichloroethane	79-00-5	Ethane, 1,1,2-trichloro-	8010	0.2
			8240	5
Trichloroethylene; Trichloroethene.	79-01-6	Ethene, trichloro-	8010	1
			8240	5
Trichlorofluoromethane	75-69-4	Methane, trichlorofluoro-	8010	10
			8240	5
2,4,5-Trichlorophenol	95-95-4	Phenol, 2,4,5-trichloro-	8270	10
2,4,6-Trichlorophenol	88-06-2	Phenol, 2,4,6-trichloro-	8040	5
			8270	10
1,2,3-Trichloropropane	96-18-4	Propane, 1,2,3-trichloro-	8010	10
			8240	5
O,O,O-Triethyl phosphorothioate	126-68-1	Phosphorothioic acid, O,O,O-triethyl ester	8270	10
sym-Trinitrobenzene	99-35-4	Benzene, 1,3,5-trinitro-	8270	10
Vanadium	(Total)	Vanadium	6010	80
			7910	2,000
			7911	40
Vinyl acetate	108-05-4	Acetic acid, ethenyl ester	8240	5
Vinyl chloride	75-01-4	Ethene, chloro-	8010	2
			8240	10

GROUND-WATER MONITORING LIST [1]—Continued

Common name [2]	CAS RN [3]	Chemical abstracts service index name [4]	Suggested methods [5]	PQL (µg/L) [6]
Xylene (total)	1330–20–7	Benzene, dimethyl-	8020 8240	5 5
Zinc	(Total)	Zinc	6010 7950	20 50

[1] The regulatory requirements pertain only to the list of substances; the right hand columns (Methods and PQL) are given for informational purposes only. See also footnotes 5 and 6.

[2] Common names are those widely used in government regulations, scientific publications, and commerce; synonyms exist for many chemicals.

[3] Chemical Abstracts Service registry number. Where "Total" is entered, all species in the ground water that contain this element are included.

[4] CAS index names are those used in the 9th Cumulative Index.

[5] Suggested methods refer to analytical procedure numbers used in the EPA publication, SW–846, "Test Methods for Evaluating Solid Waste", Third Edition. Analytical details can be found in SW–846 and in documentation on file at the Agency. The packed column gas chromatography methods 8010, 8020, 8030, 8040, 8060, 8080, 8090, 8110, 8120, 8140, 8150, 8240, and 8250 were promulgated methods through Update IIB of SW–846 and, as of Update III, the Agency has replaced these methods with "capillary column GC methods", as the suggested methods.

[6] Practical Quantitation Limits (PQLs) are the lowest concentrations of analytes in ground waters that can be reliably determined within specified limits of precision and accuracy by the indicated methods under routine laboratory operating conditions. The PQLs listed are generally stated to one significant figure. CAUTION: The PQL values in many cases are based only on a general estimate for the method and not on a determination for individual compounds; PQLs are not a part of the regulation.

[7] Polychlorinated biphenyls (CAS RN 1336–36–3); this category contains congener chemicals, including constituents of Aroclor-1016 (CAS RN 12674–11–2), Aroclor-1221 (CAS RN 11104–28–2), Aroclor-1232 (CAS RN 11141–16–5), Aroclor-1242 (CAS RN 53469–21–9), Aroclor-1248 (CAS RN 12672–29–6), Aroclor-1254 (CAS RN 11097–69–1), and Aroclor-1260 (CAS RN 11096–82–5). The PQL shown is an average value for PCB congeners.

[8] This category contains congener chemicals, including tetrachlorodibenzo-p-dioxins (see also 2,3,7,8-TCDD), pentachlorodibenzo-p-dioxins, and hexachlorodibenzo-p-dioxins. The PQL shown is an average value for PCDD congeners.

[9] This category contains congener chemicals, including tetrachlorodibenzofurans, pentachlorodibenzofurans, and hexachlorodibenzofurans. The PQL shown is an average value for PCDF congeners.

[52 FR 25947, July 9, 1987, as amended at 62 FR 32462, June 13, 1997]

PART 265—INTERIM STATUS STANDARDS FOR OWNERS AND OPERATORS OF HAZARDOUS WASTE TREATMENT, STORAGE, AND DISPOSAL FACILITIES

Subpart A—General

Sec.
265.1 Purpose, scope, and applicability.
265.2—265.3 [Reserved]
265.4 Imminent hazard action.

Subpart B—General Facility Standards

265.10 Applicability.
265.11 Identification number.
265.12 Required notices.
265.13 General waste analysis.
265.14 Security.
265.15 General inspection requirements.
265.16 Personnel training.
265.17 General requirements for ignitable, reactive, or incompatible wastes.
265.18 Location standards.
265.19 Construction quality assurance program.

Subpart C—Preparedness and Prevention

265.30 Applicability.
265.31 Maintenance and operation of facility.
265.32 Required equipment.
265.33 Testing and maintenance of equipment.
265.34 Access to communications or alarm system.
265.35 Required aisle space.
265.36 [Reserved]
265.37 Arrangements with local authorities.

Subpart D—Contingency Plan and Emergency Procedures

265.50 Applicability.
265.51 Purpose and implementation of contingency plan.
265.52 Content of contingency plan.
265.53 Copies of contingency plan.
265.54 Amendment of contingency plan.
265.55 Emergency coordinator.
265.56 Emergency procedures.

Subpart E—Manifest System, Recordkeeping, and Reporting

265.70 Applicability.
265.71 Use of manifest system.
265.72 Manifest discrepancies.
265.73 Operating record.
265.74 Availability, retention, and disposition of records.
265.75 Biennial report.
265.76 Unmanifested waste report.
265.77 Additional reports.

ATTACHMENT C

BATTERY MATERIAL SAFETY DATA SHEETS

ENERGIZER POWER SYSTEMS -- NICKEL CADMIUM BATTERY, DRY CELL - BATTERY,STORAGE NICK
MATERIAL SAFETY DATA SHEET
NSN: 6140009213670
Manufacturer's CAGE: ENERG
Part No. Indicator: A
Part Number/Trade Name: NICKEL CADMIUM BATTERY, DRY CELL

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General Information

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Item Name: BATTERY,STORAGE NICKEL-CADMIUM,SEALED
Company's Name: ENERGIZER POWER SYSTEMS
Company's P. O. Box: 147114
Company's City: GANESVILLE
Company's State: FL
Company's Country: US
Company's Zip Code: 32514-7114
Company's Emerg Ph #: 904-482-3911
Company's Info Ph #: 904-482-3911
Distributor/Vendor # 1: GENERAL ELECTRIC (904-377-4466)
Distributor/Vendor # 1 Cage: 0YUJ0
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 003
Status: SE
Date MSDS Prepared: 06JUL90
Safety Data Review Date: 03MAY94
Supply Item Manager: CX
MSDS Preparer's Name: S.Z.MANEDORF & ASSOC INC
MSDS Serial Number: BTHHZ
Specification Number: MIL-B-55130/2
Hazard Characteristic Code: N1
Unit Of Issue: EA

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Ingredients/Identity Information

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Proprietary: NO
Ingredient: NICKEL (SARA III)
Ingredient Sequence Number: 01
NIOSH (RTECS) Number: QR5950000
CAS Number: 7440-02-0
OSHA PEL: 1 MG/M3
ACGIH TLV: 1 MG/M3; 9394
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: CADMIUM (AS CD);(SARA III)
Ingredient Sequence Number: 02
NIOSH (RTECS) Number: EU9800000
CAS Number: 7440-43-9
OSHA PEL: 0.2 MG/M3 DUST; Z-2
ACGIH TLV: 0.01 MG/M3 DUST;9394
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: COBALT (SARA III),DUST AND FUME
Ingredient Sequence Number: 03
NIOSH (RTECS) Number: GF8750000
CAS Number: 7440-48-4
OSHA PEL: 0.1 MG/M3
ACGIH TLV: 0.05 MG/M3;DUST 9394
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: POTASSIUM HYDROXIDE (SARA III)

Ingredient Sequence Number: 04
NIOSH (RTECS) Number: TT2100000
CAS Number: 1310-58-3
OSHA PEL: C, 2 MG/M3
ACGIH TLV: C 2 MG/M3; 9394
Other Recommended Limit: NONE RECOMMENDED

Physical/Chemical Characteristics

Appearance And Odor: SEALED METALLIC CYLINDER WITH NO ODOR.
Boiling Point: N/A
Melting Point: N/A
Vapor Pressure (MM Hg/70 F): N/A
Vapor Density (Air=1): N/A
Specific Gravity: N/A
Decomposition Temperature: N/A
Evaporation Rate And Ref: N/A
Solubility In Water: N/A
Percent Volatiles By Volume: N/A
Viscosity: N/A
pH: N/A
Corrosion Rate (IPY): N/A
Autoignition Temperature: N/A

Fire and Explosion Hazard Data

Flash Point: N/A
Lower Explosive Limit: N/A
Upper Explosive Limit: N/A
Extinguishing Media: DRY CHEMICAL, FOAM, CARBON DIOXIDE.
Special Fire Fighting Proc: USE NIOSH-APPROVED SELF-CONTAINED BREATHING APPARATUS, OPERATING IN THE POSITIVE PRESSURE MODE AND FULL FIRE FIGHTING PROTECTIVE CLOTHING.
Unusual Fire And Expl Hazrds: CAMIUN, NICKLE, AND COBALT FUMES MAY BE EVOLVED IN FIRE SITUATION.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): AVOID PROLONGED EXPOSURE TO HUMID CONDITIONS OR EXCESSIVE HEAT.
Materials To Avoid: NOT APPLICABLE.
Hazardous Decomp Products: OXIDES OF CARBON AND METALLIC OXIDES UPON BURNING (CADMIUM, NICKEL, COBALT).
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NOT RELEVANT.

Health Hazard Data

LD50-LC50 Mixture: NOT APPLICABLE.
Route Of Entry - Inhalation: NO
Route Of Entry - Skin: NO
Route Of Entry - Ingestion: NO
Health Haz Acute And Chronic: ACUTE/CHRONIC:NONE KNOWN OR EXPECTED UNDER IRRIT. EYE:CORROSIVE EYE DAMAGE.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: YES
Explanation Carcinogenicity: CADMIUM & NICKEL & THEIR CMPS CONSIDERED POTENTIAL HUMAN CARCINOGENS BY NTP & IARC. OSHA HAS PROPOSED CADMIUM/CMP CARCIN
Signs/Symptoms Of Overexp: SKIN:REDNESS, DISCOMFORT. EYE:CORROSIVE DAMAGE.
Med Cond Aggravated By Exp: NONE SPECIFIED BY MANUFACTURER.
Emergency/First Aid Proc: SKIN:WASH AREA THOROUGHLY WITH SOAP AND WATER.

GET MED ATTN IF IRRIT DEVELOPS. EYE:FLUSH THOROUGHLY WITH RUNNING WATER FOR AT LEAST 15MINS HOLDING EYELIDS OPEN. GET PROMPT MED ATTN.

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Precautions for Safe Handling and Use

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Steps If Matl Released/Spill: RECOVER PRODUCT AND PLACE IN SUITABLE CONTAINER FOR REUSE OR DISPOSAL. AVOID CONDITIONS THAT MAY CAUSE SHORT-CIRCUITING. IF BATTERY CONTENTS ARE RELEASED DO NOT TOUCH SPILLED MATL. TAKE UP W/SAND OR OTHER ABSORBENT. PLACE IN CONTAINER FOR DISPOSAL.
Neutralizing Agent: NONE SPECIFIED BY MANUFACTURER.
Waste Disposal Method: DO NOT INCINERATE. DISPOSE OF IAW LOCAL, STATE AND FEDERAL REGULATIONS.
Precautions-Handling/Storing: STORE IN A COOL, DRY PLACE. DO NOT PUNCTURE, MUTILATE OR INCINERATE. DO NOT SHORT-CIRCUIT.
Other Precautions: PRODUCT MAY BURST RELEASING HAZARDOUS MATERIAL. MAY CAUSE THERMAL BURN OR SERVE AS IGNITION SOURCE IN FLAMMABLE OR EXPLOSIVE ATMOSPHERE.

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Control Measures

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Respiratory Protection: NONE REQUIRED UNDER NORMAL CONDITIONS OF USE.
Ventilation: NONE REQUIRED UNDER NORMAL CONDITIONS OF USE.
Protective Gloves: NONE REQUIRED UNDER NORMAL CONDITIONS.
Eye Protection: SAFETY GLASSES.
Other Protective Equipment: NONE SPECIFIED BY MANUFACTURER.
Work Hygienic Practices: NONE SPECIFIED BY MANUFACTURER.
Suppl. Safety & Health Data: PRODUCT IS AN "ARTICLE". THE PHYSICAL FORM OF PRODUCT PRECLUDES EXPOSURE. GE HAVE SOLD SOME BATTERIES TO EVERYREADY.

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Transportation Data

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Trans Data Review Date: 94123
DOT PSN Code: BQL
DOT Proper Shipping Name: BATTERIES, DRY, CONTAINING POTASSIUM HYDROXIDE SOLID
DOT Class: 8
DOT ID Number: UN3028
DOT Pack Group: III
DOT Label: CORROSIVE
IMO PSN Code: BWA
IMO Proper Shipping Name: BATTERIES, DRY, CONTAINING POTASSIUM HYDROXIDE, SOLID,
IMO Regulations Page Number: 8119
IMO UN Number: 3028
IMO UN Class: 8
IMO Subsidiary Risk Label: -
IATA PSN Code: CZJ
IATA UN ID Number: 3028
IATA Proper Shipping Name: BATTERIES, DRY, CONTAINING POTASSIUM HYDROXIDE, SOLID
IATA UN Class: 8
IATA Label: CORROSIVE
AFI PSN Code: CZJ
AFI Prop. Shipping Name: BATTERIES, DRY, CONTAINING POTASSIUM HYDROXIDE, SOLID
AFI Class: 8
AFI ID Number: UN3028
AFI Pack Group: III
AFI Basic Pac Ref: 12-7

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Disposal Data

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Label Data

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Label Required: YES
Technical Review Date: 03MAY94
Label Status: F
Common Name: NICKEL CADMIUM BATTERY, DRY CELL
Chronic Hazard: NO
Signal Word: WARNING!
Acute Health Hazard-Moderate: X
Contact Hazard-Moderate: X
Fire Hazard-None: X
Reactivity Hazard-None: X
Special Hazard Precautions: ACUTE/CHRONIC:NONE KNOWN OR EXPECTED UNDER WASH AREA THOROUGHLY WITH SOAP AND WATER. GET MED ATTN IF IRRIT DEVELOPS. EYE:FLUSH THOROUGHLY WITH RUNNING WATER FOR AT LEAST 15MINS HOLDING EYELIDS OPEN. GET PROMPT MED ATTN. STORE IN COOL, DRY PLACE. DO NOT PUNCTURE, MUTILATE, OR INCINERATE. DO NOT SHORT-CIRCUIT.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: ENERGIZER POWER SYSTEMS
Label P.O. Box: 147114
Label City: GANESVILLE
Label State: FL
Label Zip Code: 32514-7114
Label Country: US
Label Emergency Number: 904-482-3911

ALCAD -- BATT,WET,FILL W-ALKALI,8,UN2795,PGIII(NICD BATT); - BATTERY SHORAGE,NICKE
MATERIAL SAFETY DATA SHEET
NSN: 6140000687356
Manufacturer's CAGE: 0E9S8
Part No. Indicator: A
Part Number/Trade Name: BATT,WET,FILL W/ALKALI,8,UN2795,PGIII(NICD BATT);
SAFT MP702T

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General Information

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Item Name: BATTERY SHORAGE,NICKEL-CADMIUM,CHARGED,SEALED CELL
Company's Name: ALCAD INC
Company's Street: 73 DEFCO PARK RD
Company's City: NORTH HAVEN
Company's State: CT
Company's Country: US
Company's Zip Code: 06473
Company's Emerg Ph #: 203-234-8333/800-424-9300(CHEMTREC)
Company's Info Ph #: 203-234-8333
Safety Data Action Code: A
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 003
Status: SE
Date MSDS Prepared: 31JAN95
Safety Data Review Date: 28MAY98
Supply Item Manager: CX
MSDS Serial Number: CGWZD
Specification Number: UNKNOWN
Hazard Characteristic Code: C2
Unit Of Issue: EA
Unit Of Issue Container Qty: 1 EACH
Type Of Container: BATTERY
Net Unit Weight: UNKNOWN

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Ingredients/Identity Information

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Proprietary: NO
Ingredient: CADMIUM (SARA 313) (CERCLA) PLUS INGRED #2
Ingredient Sequence Number: 01
Percent: 6
Ingredient Action Code: A
NIOSH (RTECS) Number: EU9800000
CAS Number: 7440-43-9
OSHA PEL: 0.2 MG/M3 DUST; Z-2
ACGIH TLV: 0.01 MG/M3 DUST;9596
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: CADMIUM HYDROXIDE PLUS INGRED #1
Ingredient Sequence Number: 02
Percent: SEE #1
Ingredient Action Code: A
NIOSH (RTECS) Number: 1000973CH
CAS Number: 21041-95-2
OSHA PEL: 0.05 MG CD/M3
ACGIH TLV: 0.05 MG CD/M3
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: NICKEL (SARA 313) (CERCLA) PLUS INGRED #4
Ingredient Sequence Number: 03
Percent: 9
Ingredient Action Code: A

NIOSH (RTECS) Number: QR5950000
CAS Number: 7440-02-0
OSHA PEL: 1 MG/M3
ACGIH TLV: 1 MG/M3; 9596
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: NICKEL HYDROXIDE (SARA 313) (CERCLA) PLUS INGRED #3
Ingredient Sequence Number: 04
Percent: SEE #3
Ingredient Action Code: A
NIOSH (RTECS) Number: QR7040000
CAS Number: 12054-48-7
OSHA PEL: 1 MG(NI)/M3
ACGIH TLV: 0.1 MG(NI)/M3; 9596
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: POTASSIUM HYDROXIDE (CERCLA); KOH; CAUSTIC POTASH
Ingredient Sequence Number: 05
Percent: 10
Ingredient Action Code: A
NIOSH (RTECS) Number: TT2100000
CAS Number: 1310-58-3
OSHA PEL: C 2 MG/M3
ACGIH TLV: C 2 MG/M3; 9596
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: LITHIUM HYDROXIDE; LIOH; LITHIA
Ingredient Sequence Number: 06
Percent: 0.5
Ingredient Action Code: A
NIOSH (RTECS) Number: OJ6307070
CAS Number: 1310-65-2
OSHA PEL: NOT ESTABLISHED
ACGIH TLV: NOT ESTABLISHED
Other Recommended Limit: NONE RECOMMENDED
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Physical/Chemical Characteristics

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Appearance And Odor: RECTANGULAR PLASTIC CNTNR ENCLOSING NICKEL & CADMIUM ELECTORDES & KOH ELECTROLTY
Boiling Point: NA
Melting Point: NA
Vapor Pressure (MM Hg/70 F): NA
Vapor Density (Air=1): NA
Specific Gravity: NA
Decomposition Temperature: NP
Evaporation Rate And Ref: NA
Solubility In Water: NA
Percent Volatiles By Volume: NP
Viscosity: NP
pH: NP
Corrosion Rate (IPY): NP
Autoignition Temperature: NP
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Fire and Explosion Hazard Data

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Flash Point: NA
Flash Point Method: NA
Lower Explosive Limit: NA
Upper Explosive Limit: NA
Extinguishing Media: SUITABLE FOR SURROUNDING MATERIAL.

Special Fire Fighting Proc: WEAR FULL PROTECTIVE CLOTHING.USE NIOSH/ MSHA-APPROVED POSITIVE PRESSURE SELF-CONTAINED BREATHING APPARATUS.
Unusual Fire And Expl Hazrds: TOXIC CADMIUM OXIDE FUMES MAY BE RELEASED DURING BURNING.POTASSIUM HYDROXIDE RXS VIOLENTLY W/ORG CHEMS,ESPECIALLY NITROCARBONS & CHLOROCARBONS,METALS (ZN,AL,SN)

Reactivity Data

Stability: YES
Cond To Avoid (Stability): NONE SPECIFIED BY MANUFACTURER.
Materials To Avoid: DONT ALLOW KOH TO CONTACT ORGANIC CHEMS,ACIDS OR METALS SUCH AS ZINC,ALUMINUM,TIN.EXPO TO AIR FORMS POTASSIUM CARBONATE.
Hazardous Decomp Products: TOXIC CADMIUM OXIDE FUMES MAY BE RELEASED IF INCINERATED.FLAMM HYDROGEN GAS PRODUCED DURING CHARGING.
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NOT APPLICABLE

Health Hazard Data

LD50-LC50 Mixture: NONE SPECIFIED BY MANUFACTURER.
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: ACUTE EXPO:KOH-BURNS TO TISSUE CONTACTED; NICKEL-POSSIBLE CONTACT DERMATITIS.CHRONIC EXPO:CADMIUM-POSSIBLE LUNG OR KIDNEY DAMAGE.NICKEL-POSSIBLE LUNG DAMAGE.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: YES
Explanation Carcinogenicity: PER MSDS:CARCINOGENICITY-CD,NI.NTP/IARC/ OSHA:YES.
Signs/Symptoms Of Overexp: BURNS TO TISSUE CONTACTED, CONTACT DERMATITIS, LUNG/KIDNEY DAMAGE.
Med Cond Aggravated By Exp: INDIVIDUALS W/PRE-EXISTING DISEASE OF LUNGS MAY HAVE INCREASED SUSCEPTIBILITY TO TOXICITY OF EXCESSIVE EXPOSURES.
Emergency/First Aid Proc: IF KOH ELECTROLYTE SPILLED IMMED FLUSH EYE/ SKIN FOR @LEAST 15MINS WHILE REMOVING CONTAM CLOTH/SHOES.MILD ACID MAY BE USED TO NEUTRALIZE KOH ON SKIN.IN ALL CASES CONTACT PHYSICIAN.IF SWALLOWED DO NOT INDUCE VOMITING.DRINK LARGE QUANTITIES OF WATER OR FRUIT JUICE. CONTACT PHYSICIAN.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: IF KOH ELECTROLYTE SPILLED COMPLY W/LOCAL, STATE AND FEDERAL REGULATIONS ON REPORTING RELEASES,CONTAINING SILLS/ REMEDIATION.CONTAIN SPILL TO SMALLEST POSSIBLE AREA & ABSORB AS APPROPRIATE.
Neutralizing Agent: WEAK ACID SOLUTIONS.
Waste Disposal Method: COMPLY W/LOCAL, STATE AND FEDERAL REGULATIONS.IF APPROVED NEURALIZE W/WEAK ACID SOLUTIONS AND TRANSFER TO WASTE TREATMENT SYSTEM.
Precautions-Handling/Storing: AVOID ROUGH HNDLG.DO NOT STORE ABOVE 140F. ALWAYS HAVE WATER SAFETY SHOWERS/EYEWASH FOUNTAINS AVAILABLE.
Other Precautions: NO SMOKING REGULATIONS MUST BE ENFORCED DUE TO HYDROGEN GENERATION DURING BATTERY CHARGING.

Control Measures

Respiratory Protection: IF INCINERATED NIOSH/MSHA APPROVED POSITIVE PRESSURE SELF-CONTAINED BREATHING APPARATUS.
Ventilation: LOCAL EXHAUST-YES-DISCHARGE OUT OF WORK AREA.
Protective Gloves: RUBBER ACID-PROOF GAUNTLET GLOVES
Eye Protection: CHEM SPLASH GOGG,FULL-LENGTH FACE SHIELD
Other Protective Equipment: RUBBER OR PLASTIC APRON & BOOTS;LONG SLEEVE

WOOL,ACRYLIC OR POLYESTER CLOTHING;ACID-PROOF SUIT AND HOOD.
Work Hygienic Practices: ALWAYS USE EXTREME CARE;WASH THOROUGHLY AFTER HANDLING.

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Transportation Data

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Transportation Action Code: A
Trans Data Review Date: 98244
DOT PSN Code: BQP
DOT Proper Shipping Name: BATTERIES, WET, FILLED WITH ALKALI
DOT Class: 8
DOT ID Number: UN2795
DOT Pack Group: III
DOT Label: CORROSIVE
IMO PSN Code: BWF
IMO Proper Shipping Name: BATTERIES,WET,FILLED WITH ALKALI
IMO Regulations Page Number: 8120
IMO UN Number: 2795
IMO UN Class: 8
IMO Subsidiary Risk Label: -
IATA PSN Code: CZR
IATA UN ID Number: 2795
IATA Proper Shipping Name: BATTERIES, WET, FILLED WITH ALKALI
IATA UN Class: 8
IATA Label: CORROSIVE
AFI PSN Code: CZR
AFI Prop. Shipping Name: BATTERIES, WET, FILLED WITH ALKALI
AFI Class: 8
AFI ID Number: UN2795
AFI Pack Group: III
AFI Special Prov: P5
AFI Basic Pac Ref: A12.5
Additional Trans Data: PER MSDS:BATTERY,WET,FILLED W/ALKALI, 8, UN 2795, PKG III.

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Disposal Data

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Label Data

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Label Required: YES
Technical Review Date: 28MAY98
Label Status: F
Common Name: BATT,WET,FILL W/ALKALI,8,UN2795,PGIII(NICD BATT);
SAFT MP702T
Chronic Hazard: YES
Signal Word: CAUTION!
Acute Health Hazard-Slight: X
Contact Hazard-Slight: X
Fire Hazard-Slight: X
Reactivity Hazard-None: X
Special Hazard Precautions: ACUTE EXPO:KOH-BURNS TO TISSUE CONTACTED; NICKEL-POSSIBLE CONTACT DERMATITIS.CHRONIC EXPO:CADMIUM-POSSIBLE LUNG OR KIDNEY DAMAGE.NICKEL-POSSIBLE LUNG DAMAGE. AVOID ROUGH HNDLG.DO NOT STORE ABOVE 140F.ALWAYS HAVE WATER SAFETY SHOWERS/EYEWASH FOUNTAINS AVAILABLE. FIRST AID: IF KOH ELECTROLYTE SPILLED IMMED FLUSH EYE/SKIN FOR @LEAST 15MINS WHILE REMOVING CONTAM CLOTH/SHOES.MILD ACID MAY BE USED TO NEUTRALIZE KOH ON SKIN.IN ALL CASES CONTACT PHYSICIAN.IF SWALLOWED DO NOT INDUCE VOMITING.DRINK LARGE QUANTITIES OF WATER OR FRUIT JUICE.CONTACT PHYSICIAN.TARGET ORGANS:TISSUE,LUNGS,KIDNEY.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: ALCAD INC

Label Street: 73 DEFCO PARK RD
Label City: NORTH HAVEN
Label State: CT
Label Zip Code: 06473
Label Country: US
Label Emergency Number: 203-234-8333/800-424-9300(CHEMTREC)

Post-it® Fax Note 7671	Date 1/3/96	# of pages ▶ 4
To Roy Williams	From BHR	
Co./Dept.	Co. Battery Assemblers	
Phone #	Phone # 516-567-8855	
Fax #	Fax #	

MATERIAL SAFETY DATA SHEET

OSHA's Hazard Communication Sheet
29 CRF 1910.1200

IDENTITY:

Alkaline Manganese Dioxide cells and batteries comprised of these cells.

MANUFACTURER:

Battery Assemblers, Inc.
211-4 Knickerbocker Avenue
Bohemia, New York 11716

EMERGENCY TELEPHONE NUMBERS:

Robert Mainzer : (516) 567-8855
Howard Eckstein: (516) 567-8855
FAX (24 hours) : (516) 567-8278

DATE PREPARED:

July 10, 1992

HAZARDOUS INGREDIENTS/IDENTITY INFORMATION:

Hazardous Components	OSHA PEL	ACGIH TLV	Other Limits	% * (Approx.)
Potassium Hydroxide (KOH)	2 mg/m^3 (C)	2 mg/m^3 (C)	--	8
Manganese Dioxide (MnO_2)	5 mg/m^3 (C)	5 mg/m^3 (C)	--	37
Zinc (Zn)	15 mg/m^3	10 mg/m^3	--	15

* Percentage of total cell weight

PHYSICAL/CHEMICAL CHARACTERISTICS:

Boiling Point::	(°)C	1320 (KOH), N/A (MnO_2), 907 (Zn) 357 (Hg)
Vapor Pressure:	(mm Hg)	N/A (KOH, MnO_2), 1 mm at 437°C (Zn), 1 mm at 126°C (Hg)
Vapor Density:	(AIR = 1)	N/A
Solubility in Water:		50% (KOH), 0% (MnO_2), 0% Zn, 0% Hg
Specific Gravity:	(H_2O = 1)	2.0 (KOH), 5.0 (MnO_2), 7.14 (Zn) 13.6 (Hg)
Melting Point:	(°C)	360 (KOH), 535 (MnO_2), 420 (Zn), -39 (Hg)
Evaporation Rate:	(Butyl Acetate = 1)	N/A (KOH, MnO_2, Zn, Hg)
Appearance and Odor:		Clear liquid (KOH), black powder (MnO_2), grey powder (Zn), amalgam with Zn (Hg)

FIRE AND EXPLOSION HAZARD DATA:

Flash Point (Method Used):	N/A	
Extinguishing Media:	N/A	
Flammable Limits:	LEL	UEL
	N/A	N/A

Special Fire Fighting Procedures:

Fire fighters should use self-contained breathing apparatus when a large number of cells are involved in a fire.

Unusual Fire and Explosion Hazards:

Cells may release toxic mercury vapors when the integrity of the cans is broken.

REACTIVITY DATA:

Stability: Stable

Conditions to Avoid: DO NOT heat, disassemble, or recharge.

Hazardous Composition or By-products:

When heated, cells may emit hazardous vapors of Hg and caustic KOH.

Hazardous Polymerization:

Will NOT occur.

Fax

To: Jerry McCracken, Code 095

Naval Surface Warfare Center, Crane Division B3260

Fax: DSN 482-4177

From . Bernie Hinkle, Code 6094

Naval Surface Warfare Center, Crane Division

Of: High Energy Power Systems (Lithium Batteries)

Fax: DSN 482-2043

Phone: DSN 482-3478 **Comm:** (812) 854-3478

Pages: 10, including this cover sheet.

D Aug 20, 1998

Mercury Battery MSDS

Jerry:

Here's a copy of MIL-B-18E slash sheet for the BA-1568/U Mercury Oxide battery. The BA-1568/U battery is made up of 10 obsolete DURACELL RM-828 cells, the MSDS is enclosed. Please inform me if approximately 240 each of these items can be disposed of through your office. If so what will be the cost. Roy Williams has said DRMO can dispose of these mercury batteries but you could probably do it cheaper. If you need a copy of MIL-SPEC-B-18E I can send a copy to you (30 pages) even though it dose'nt say much. If more info is need please contact me via phone, fax or e-mail as shown below:

Tele: DSN 482-3478

Fax: DSN 482-2043

e-mail: hinklebj@crane.navy.mil

ernie Hinkle ,Code 6094

From the desk of...

Bernie HINKLE
Electronics Technician
U.S Navy DOD
Naval Surface Warfare Center
Crane, Indiana 47522
(812) 854-3478 DSN 482-3478

Gillette Medical Evaluation Laboratories

401 Professional Drive
Gaithersburg, MD 20879
Tel 301.590.9781

MATERIAL SAFETY DATA SHEET

NAME: DURACELL MERCURIC OXIDE BATTERIES

Effective Date: 5/6/98 Rev: NA

CAS NO: Not applicable

A. — IDENTIFICATION

	%
Mercuric Oxide (21908-53-2)	35-45
Zinc (7440-66-6)	15
Manganese Dioxide (1313-13-9)	5-10
Potassium Hydroxide (35%) (1310-58-3)	5-10
Mercury (7439-97-6)	<1

Formula: Mixture

Molecular Weight: NA

Synonyms: Mercuric Oxide Cells: TR164R; TR165R; TR175; TR177; TR286; TR289; TR431B; ZM9; 303996; 304116B; RM13H6;, RM13D; RM312H6; RM312D; MP675H6; MP401H2; RM41H6; RM41D; PX32B; PX625B; PX640B; PX675B; RM400RB; D343; MP401D; RM1R; RM12R;; RM601R; RM640R; RM640RB; TR132R; TR133R; TR134R; TR135R; TR136R; TR146X; PX21

B. — PHYSICAL DATA

Boiling Point NA °F NA °C	Melting Point NA °F NA °C	Freezing Point NA °F NA °C
Specific Gravity (H_2O=1) NA	Vapor Density (air=1) NA	Vapor Pressure @ ___ °F NA mm Hg
Evaporation (Ether =1) NA	Saturation in Air (by volume@ ___ °F) NA	Autoignition Temperature ___ °F ___ °C NA
% Volatiles NA	Solubility in Water NA	pH NA

Appearance/Color: Various size batteries. Contents dark in color.

Flash Point and Test Method(s): NA

Flammable Limits in Air (% by volume): Lower NA % Upper NA %

C. — REACTIVITY

Stability: [X] stable [ ] unstable

Polymerization: [ ] may occur [X] will not occur

Conditions to Avoid: Do not heat, crush, disassemble, short circuit or recharge.

Conditions to Avoid: Not applicable

Incompatible Materials: Contents incompatible with strong oxidizing agents.

Hazardous Decomposition Products: Thermal degradation may produce hazardous fumes of mercury, zinc, manganese; hydrogen gas; caustic vapors of potassium hydroxide and other toxic by-products.

* IF MULTIPLE INGREDIENTS, INCLUDE CAS NUMBERS FOR EACH NA=NOT AVAILABLE

Footnotes: Not applicable

F. — EXPOSURE CONTROL METHODS

Engineering Controls
General ventilation under normal use conditions.

Eye Protection
None under normal use conditions. Wear safety glasses when handling leaking batteries.

Skin Protection
None under normal use conditions. Use neoprene, rubber or latex-nitrile gloves when handling leaking batteries.

Respiratory Protection
None under normal use conditions.

Other
Keep batteries away from small children.

G. — WORK PRACTICES

Handling and Storage
Store at room temperature. Avoid mechanical or electrical abuse. **DO NOT** short or install incorrectly. Batteries may explode, pyrolize or vent if disassembled, crushed, recharged or exposed to high temperatures. Install batteries in accordance with equipment instructions. Do not mix battery systems, such as alkaline and zinc carbon, in the same equipment. Replace all batteries in equipment at the same time. Do not carry batteries loose in pocket or bag.

Normal Clean Up
Not applicable

Waste Disposal Methods
No special precautions are required for small quantities. Large quantities of open batteries should be treated as hazardous waste. Dispose of in accordance with federal, state and local regulations. Do not incinerate, since batteries may explode at excessive temperatures.

GNB

Material Safety Data Shee

[redacted]

HMIS

H 0
F 3
R 2
PERSONAL PROTECTION

I PRODUCT IDENTIFICATION

Chemical/Trade name (identity used on label)	[redacted]
Chemical family/Classification	Electric Storage Battery
Company name	GNB Battery Technologies
Address	1110 Highway 110, Mendota Heights, MN 55118
Division or department	Automotive Battery Division
Telephone number	612-681-5000
CONTACT	Richard B. Crowell
Questions	Contact Environmental Development Department, 612/681-5000 or 681-5281
Transportation emergencies	CHEMTREC, 24 hours 800-424-9300

II HAZARDOUS INGREDIENTS

Material	% by Wt. or Vol.	CAS Number	Exposure Limits OSHA	Exposure Limits ACGIH
Lead	27-34	7439-92-1	.05mg/m³	.15mg/m³
Lead oxides	39-48	1309-60-0	.05mg/m³	.15mg/m³
Electrolyte (sulfuric acid and water)	11-23	7664-93-9	1.0mg/m³	1.0mg/m³
Case material: Polypropylene	6-10	9003-07-0	N/A	N/A
Hard rubber	5-10	9003-55-8	N/A	N/A
Plate separator material: Polyethylene	1-2	9002-86-2	N/A	N/A

III PHYSICAL DATA

Materials (at normal temperatures)	Electrolyte (sulfuric acid and water)
Boiling point (at 760 MM Hg)	229-248° F
Melting point	Not applicable
Specific gravity (H_2O = 1)	1.230 to 1.350
Vapor density (AIR = 1)	Greater than 1
% Volatiles by weight	Not applicable
Appearance and odor	Electrolyte (sulfuric acid and water) is a clear liquid with a sharp, penetrating, pungent odor.
Vapor pressure (mm Hg at 20C)	10
Solubility in water	100%
Evaporation rate (Butyl acetate = 1)	Less than 1

VIII SAFE HANDLING PRECAUTIONS

Hygiene practices	Wash hands thoroughly before eating, drinking or smoking after handling batteries.
Protective measures to be taken during non routine tasks including equipment maintenance	Not applicable.

SPILL OR LEAK PROCEDURES

Protective measures to be taken if material is released or spilled	Remove combustible materials and all sources of ignition. Stop flow of material and contain spill by diking with soda ash (sodium carbonate) or quick lime (calcium oxide). Carefully neutralize spill with soda ash, etc. Make certain mixture is neutral then collect residue and place in a drum or other suitable container with a label specifying "contains hazardous waste" or if uncertain call distributor regarding proper labeling procedures. Dispose of as hazardous waste. If battery is leaking, place battery in a heavy duty plastic bag. Wear acid resistant boots, face shield, chemical splash goggles and acid resistant gloves. DO NOT RELEASE UNNEUTRALIZED ACID.
Waste disposal methods	Sulfuric acid: Neutralize as described above for a spill, collect residue and place in a container labeled as containing hazardous waste. Dispose of as a hazardous waste. If uncertain about labeling procedures call your local battery distributor or contact listed on page 1 of this MSDS. DO NOT FLUSH LEAD CONTAMINATED ACID TO SEWER. Batteries: Send to lead recycling facility following applicable federal, state and local regulations. Contact GNB Environmental Development for further information on proper routine disposition of spent batteries.

IX OTHER

REGULATORY INFORMATION

NFPA hazard rating for sulfuric acid	Flammability (Red) = 0 Health (Blue) = 3 Reactivity (Yellow) = 2
US DOT identification and description for batteries and acid is as follows:	Batteries, wet, filled with acid .....UN 2794 Battery fluid, acid .....UN 2796

Lead acid batteries do not contain any ozone depleting materials.

Sulfuric acid is water reactive if concentrated. Batteries are regulated under applicable US DOT, RCRA, CERCLA and EPCRA. If you have any questions please contact the Environmental Development Department of GNB Battery Technologies at 512/881-5000 or Richard B. Crowell at 512/881-5281.

Material Safety Data Sheet

ATTN: Roy
DRMO
From

System Thermal Battery

Part Number 408360

OPTIONAL FORM 99 (7-90)

FAX TRANSMITTAL # of pages ▸ 3

To Janet McCracken	From Sam Stuart
Dept/Agency x 6153	Phone # x 5958, B34
Fax # x 4177	Fax #

NSN 7540-01-317-7368 5099-101 GENERAL SERVICES ADMINISTRATION

This MSDS was prepared by Naval Surface Warfare Center Crane Division on December 12, 1996 to allow properly designating and shipping this thermal battery.

1.0 Manufacturer's Name

System Thermal Batteries were manufactured by Catalyst Research Company (a division of Mine Safety Appliances Co.) in Baltimore, MD in 1985 and 1986. Catalyst Research was purchased by competitor Saft America (located in Cockeysville, MD, phone number is 410-771-3200) in 1990. Saft America halted most thermal battery production in 1992 and Catalyst Research does not produce thermal batteries in the United States.

2.0 Description

This battery is a percussion activated reserve thermal battery that, when activated, provides less than 30 volts for less than five minutes. Cells are constructed with lithium anodes, inorganic salt electrolyte and iron disulfide cathodes that do not provide electrical power until the battery is heated internally. Upon striking a percussion primer (at bottom of battery), the battery will activate and the external case temperature will rise by several hundred degrees Fahrenheit within approximately 30 seconds.

Physically, the battery is a 1.6 inch (3.9 cm) long metal cylinder with a diameter of 1.2 inches (2.9 cm). The percussion primer that activates the battery is located on the bottom of the steel case. Two electrical terminal posts are located at the battery header.

3.0 Contents

The battery is hermetically sealed within a stainless steel case and the chemical contents are not exposed. Hazardous (or otherwise regulated) materials are listed in the following table. Other components inside the battery include ceramic fiber thermal insulation, fiberglass tape, steel straps, glass seals and a hermetically sealed case made of 304 stainless steel. The total battery weight is approximately 78 grams.

5.0 Hazards —continued

- There is no chemical reactivity hazard associated with the sealed battery. A ruptured battery or any contents should not be allowed to contact moisture or liquids.
- Other than a high case temperature if activated, there are no other health hazards associated with the sealed battery. A ruptured thermal battery should be handled with protective gloves and eyewear if the case is cool. Avoid direct contact with cell materials.
- Other than a high case temperature if activated, there are no other fire and explosion hazards associated with the sealed battery. A ruptured battery may expose heat paper and pellets, which are electrostatic shock sensitive and will burn if exposed to an ignition source. Exposed primer material is also shock, electrostatic shock and heat sensitive.
- If the battery is subjected to fire or other conditions resulting in extremely high temperatures, it may rupture and vent cell contents and combustion products (smoke, flame and various sulfur compounds).

6.0 Spill/Leakage

- Inactive System Thermal Batteries do not contain any liquids.
- If battery case becomes severely damaged and cell contents are exposed, place the battery in a sealed steel container. Do not expose contents to ignition sources or water (lithium metal will react violently with water).

7.0 Transportation Information

- System Thermal Batteries may be shipped as **unregulated material** provided they are packaged to prevent accidental activation.
- To prevent accidental activation, the primer end of the battery must be protectively wrapped or capped to keep hard objects from striking the primer head.
- The terminal end of the battery should be wrapped or capped to prevent shorting between the terminals. Arcing could result if the battery were inadvertently activated.
- Shipments of the System Thermal Batteries must be accompanied by the Department of Transportation competent authority letter that designates this battery's shipping class.

8.0 Disposal

Once activated, no reuse is possible. System thermal batteries must be disposed of by an EPA and state approved facility that is experienced with handling these devices. **Do not throw thermal batteries into the trash!**

ATTACHMENT D

SOIL SCREENING LEVELS

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
83-32-9	Acenaphthene	4700	---	570	NA	1.1E+02	---	3.7E+02
208-96-8	Acenaphthylene	---	---	---	---	---	---	---
67-64-1	Acetone	7800	100000	16	NA	8.8E+03	---	6.1E+02
75-05-8	Acetonitrile; Methyl cyanide	---	---	---	2.2E+02	1.2E+03	---	7.1E+01
98-86-2	Acetophenone	---	---	---	4.9E-01	1.6E+00	---	4.2E-02
53-96-3	2-Acetylaminofluorene; 2-AAF	---	---	---	---	---	---	---
107-02-8	Acrolein	---	---	---	1.0E-01	3.4E-01	---	4.2E-02
107-13-1	Acrylonitrile	---	---	---	1.9E-01	4.7E-01	---	3.7E+00
309-00-2	Aldrin	0.04	3	0.5	NA	1.1E-01	---	4.0E-03
107-05-1	Allyl chloride	---	---	---	3.2E+03	3.3E+04	---	1.8E+03
92-67-1	4-Aminobiphenyl	---	---	---	---	---	---	---
62-53-3	Aniline	---	---	---	1.9E+01	2.0E+02	---	1.1E+01
120-12-7	Anthracene	23000	---	12000	NA	5.7E+00	---	1.8E+03
7440-36-0	Antimony	31	---	5	NA	6.8E+02	---	1.5E+01
140-57-8	Aramite	---	---	---	1.8E+01	7.6E+01	---	2.7E+00
7440-38-2	Arsenic	0.4	750	29	NA	2.4E+00	---	4.5E-02
7440-39-3	Barium	5500	690000	1600	NA	1.0E+05	---	2.6E+03
71-43-2	Benzene	22	0.8	0.03	NA	1.4E+00	5	3.9E-01
56-55-3	Benzo[a]anthracene; Benzanthracene	0.9	---	2	NA	2.6E+00	---	9.2E-02
205-99-2	Benzo[b]fluoranthene	0.9	---	5	NA	2.6E+00	---	9.2E-02
207-08-9	Benzo[k]fluoranthene	9	---	49	NA	2.6E+01	---	9.2E-01
191-24-2	Benzo[g,h,i]perylene	---	---	---	---	---	---	---
50-32-8	Benzo[a]pyrene	0.09	---	8	NA	2.6E-01	0.2	9.2E-03
100-51-6	Benzyl alcohol	---	---	---	2.0E+04	1.0E+05	---	1.1E+04
7440-41-7	Beryllium	0.1	1300	63	NA	1.1E+00	---	1.6E-02
319-84-6	alpha-BHC	0.1	0.8	0.0005	NA	3.0E-01	---	1.1E-02
319-85-7	beta-BHC	0.4	---	0.003	NA	1.1E+00	---	3.7E-02
319-86-8	delta-BHC	---	---	---	---	---	---	---
58-89-9	gamma-BHC; Lindane	0.5	---	0.009	NA	1.5E+00	0.2	5.2E-02
111-91-1	Bis(2-chloroethoxy)methane	---	---	---	---	---	---	---
111-44-4	Bis(2-chloroethyl)ether	0.6	0.2	0.0004	NA	9.7E-02	---	9.8E-03

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
	Bis(2-chloro-1-methylethyl) ether; 2,2-							
108-60-1	Dichlorodiisopropyl ether	---	---	---	6.3E+00	2.7E+01	---	9.6E-01
117-81-7	Bis(2-ethylhexyl) phthalate	46	31000	3600	NA	1.4E+02	---	4.8E+00
75-27-4	Bromodichloromethane	10	3000	0.6	NA	1.4E+00	---	1.8E-01
75-25-2	Bromoform; Tribromomethane	81	53	0.8	NA	2.4E+02	---	8.5E+00
101-55-3	4-Bromophenyl phenyl ether	---	---	---	---	---	---	---
85-68-7	Butyl benzyl phthalate; Benzyl butyl phthalate	16000	930	930	NA	9.3E+02	---	7.3E+03
7440-43-9	Cadmium	78	1800	8	NA	8.5E+02	---	1.8E+01
75-15-0	Carbon disulfide	7800	720	32	NA	2.4E+01	---	2.1E+01
56-23-5	Carbon tetrachloride	5	0.3	0.07	NA	5.0E-01	---	1.7E-01
57-74-9	Chlordane	0.5	20	10	NA	1.5E+00	---	5.2E-02
106-47-8	p-Chloroaniline	310	---	0.7	NA	2.7E+03	---	1.5E+02
108-90-7	Chlorobenzene	1600	130	1	NA	2.2E+02	---	3.9E+01
510-15-6	Chlorobenzilate	---	---	---	1.6E+00	7.1E+00	---	2.5E-01
59-50-7	p-Chloro-m-cresol	---	---	---	---	---	---	---
75-00-3	Chloroethane; Ethyl chloride	---	---	---	1.1E+03	1.6E+03	---	7.1E+02
67-66-3	Chloroform	100	0.3	0.6	NA	5.3E-01	---	1.6E-01
91-58-7	2-Chloronaphthalene	---	---	---	1.1E+02	1.1E+02	---	4.9E+02
95-57-8	2-Chlorophenol	390	53000	4	NA	3.7E+02	---	3.8E+01
7005-72-3	4-Chlorophenyl phenyl ether	---	---	---	---	---	---	---
126-99-8	Chloroprene	---	---	---	3.6E+00	1.2E+01	---	1.4E+01
7440-47-3	Chromium (total)	390	270	38	NA	4.5E+02	---	---
218-01-9	Chrysene	88	---	160	NA	7.2E+00	---	9.2E+00
7440-48-4	Cobalt	---	---	---	4.6E+03	9.7E+04	---	2.2E+03
7440-50-8	Copper	---	---	---	2.8E+03	6.3E+04	---	1.4E+03
108-39-4	m-Cresol	---	---	---	3.3E+03	3.4E+04	---	1.8E+03
95-48-7	o-Cresol	3900	---	15	NA	3.4E+04	---	1.8E+03
106-44-5	p-Cresol	---	---	---	3.3E+02	3.4E+03	---	1.8E+02
57-12-5	Cyanide	1600	---	40	NA	1.4E+04	---	7.3E+02
94-75-7	2,4-D; 2,4-Dichlorophenoxyacetic acid	---	---	---	6.5E+02	6.8E+03	70	3.7E+02
72-54-8	4,4'-DDD	3	---	16	NA	7.9E+00	---	2.8E-01

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
72-55-9	4,4'-DDE	2	---	54	NA	5.6E+00	---	2.0E-01
50-29-3	4,4'-DDT	2	---	32	NA	5.6E+00	---	2.0E-01
2303-16-4	Diallate	---	---	---	7.3E+00	3.1E+01	---	1.1E+00
53-70-3	Dibenz[a,h]anthracene	0.09	---	2	NA	2.6E-01	---	9.2E-03
132-64-9	Dibenzofuran	---	---	---	1.4E+02	1.4E+02	---	2.4E+01
124-48-1	Dibromochloromethane; Chlorodibromomethane	8	1300	0.4	NA	2.3E+01	---	1.0E+00
96-12-8	1,2-Dibromo-3-chloropropane; DBCP	---	---	---	3.2E-01	1.4E+00	0.2	4.8E-02
106-93-4	1,2-Dibromoethane; Ethylene	---	---	---	4.9E-03	2.0E-02	---	7.6E-04
84-74-2	Di-n-butyl phthalate	7800	2300	2300	NA	6.8E+04	---	3.7E+03
95-50-1	o-Dichlorobenzene	7000	560	17	NA	7.0E+02	600	3.7E+02
541-73-1	m-Dichlorobenzene	---	---	---	5.0E+02	8.6E+02	---	1.8E+02
106-46-7	p-Dichlorobenzene	27	---	2	NA	8.5E+00	75	4.7E-01
91-94-1	3,3-Dichlorobenzidine	1	---	0.007	NA	4.2E+00	---	1.5E-01
110-57-6	trans-1,4-Dichloro-2-butene	---	---	---	---	---	---	---
75-71-8	Dichlorodifluoromethane	---	---	---	9.4E+01	3.1E+02	---	3.9E+02
75-34-3	1,1-Dichloroethane	7800	1300	23	NA	1.7E+03	---	8.1E+02
107-06-2	1,2-Dichloroethane	7	0.4	0.02	NA	5.5E-01	5	1.2E-01
75-35-4	1,1-Dichloroethylene	1	0.07	0.06	NA	8.0E-02	7	4.6E-02
156-60-5	trans-1,2-Dichloroethylene	1600	3100	0.7	NA	2.7E+02	100	1.2E+02
120-83-2	2,4-Dichlorophenol	230	---	1	NA	2.0E+03	---	1.1E+02
87-65-0	2,6-Dichlorophenol	---	---	---	---	---	---	---
78-87-5	1,2-Dichloropropane	9	15	0.03	NA	6.8E-01	5	1.6E-01
542-75-6	1,3-Dichloropropene (mixture)	4	0.1	0.004	NA	5.5E-01	---	8.1E-02
10061-01-5	cis-1,3-Dichloropropene [d]	---	---	---	---	---	---	---
10061-02-6	trans-1,3-Dichloropropene [d]	---	---	---	---	---	---	---
60-57-1	Dieldrin	0.04	1	0.004	NA	1.2E-01	---	4.2E-03
84-66-2	Diethyl phthalate	63000	2000	470	NA	1.0E+05	---	2.9E+04
297-97-2	O,O-Diethyl O-2-pyrazinyl phosphorothioate; Thionazin	---	---	---	---	---	---	---
60-51-5	Dimethoate	---	---	---	1.3E+01	1.4E+02	---	7.3E+00
60-11-7	p-(Dimethylamino)azobenzene	---	---	---	---	---	---	---

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
57-97-6	7,12-Dimethylbenz[a]anthracene	---	---	---	---	---	---	---
119-93-7	3,3-Dimethylbenzidine	---	---	---	4.8E-02	2.1E-01	---	7.3E-03
122-09-8	alpha, alpha-Dimethylphenethylamine	---	---	---	---	---	---	---
105-67-9	2,4-Dimethylphenol	1600	---	9	NA	1.4E+04	---	7.3E+02
131-11-3	Dimethyl phthalate	---	---	---	1.0E+05	1.0E+05	---	3.7E+05
99-65-0	m-Dinitrobenzene	---	---	---	6.5E+00	6.8E+01	---	3.7E+00
534-52-1	4,6-Dinitro-o-cresol	---	---	---	---	---	---	---
51-28-5	2,4-Dinitrophenol	160	---	0.3	NA	1.4E+03	---	7.3E+01
121-14-2	2,4-Dinitrotoluene	0.9	---	0.0008	NA	1.4E+03	---	7.3E+01
606-20-2	2,6-Dinitrotoluene	0.9	---	0.0007	NA	6.8E+02	---	3.7E+01
88-85-7	Dinoseb; DNBP; 2-sec-Butyl- 4,6-dinitrophenol	---	---	---	6.5E+01	6.8E+02	7	3.7E+01
117-84-0	Di-n-octyl phthalate	1600	10000	10000	NA	1.0E+04	---	7.3E+02
123-91-1	1,4-Dioxane	---	---	---	4.0E+01	1.7E+02	---	6.1E+00
122-39-4	Diphenylamine	---	---	---	1.6E+03	1.7E+04	---	9.1E+02
298-04-4	Disulfoton	---	---	---	2.6E+00	2.7E+01	---	1.5E+00
115-29-7	Endosulfan (mixture)	470	---	1.80E+01	---	4.1E+03	---	2.2E+02
959-98-8	Endosulfan I [d]	---	---	---	---	---	---	---
33213-65-9	Endosulfan II [d]	---	---	---	---	---	---	---
1031-07-8	Endosulfan sulfate	---	---	---	---	---	---	---
72-20-8	Endrin	23	---	1	NA	2.0E+02	2	1.1E+01
7421-93-4	Endrin aldehyde	---	---	---	---	---	---	---
100-41-4	Ethylbenzene	7800	400	13	NA	2.3E+02	700	1.3E+03
97-63-2	Ethyl methacrylate	---	---	---	1.4E+02	1.4E+02	---	5.5E+02
62-50-0	Ethyl methanesulfonate	---	---	---	---	---	---	---
52-85-7	Famphur	---	---	---	---	---	---	---
206-44-0	Fluoranthene	3100	---	4300	NA	2.7E+04	---	1.5E+03
86-73-7	Fluorene	3100	---	560	NA	9.0E+01	---	2.4E+02
76-44-8	Heptachlor	0.1	4	23	NA	4.2E-01	0.4	1.5E-02
1024-57-3	Heptachlor epoxide	0.07	5	0.7	NA	2.1E-01	0.2	7.4E-03
118-74-1	Hexachlorobenzene	0.4	1	2	NA	1.2E+00	1	4.2E-02

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
87-68-3	Hexachlorobutadiene	8	8	2	NA	2.4E+01	---	8.6E-01
77-47-4	Hexachlorocyclopentadiene	550	10	400	NA	4.6E+03	50	2.6E+02
67-72-1	Hexachloroethane	46	55	0.5	NA	1.4E+02	---	4.8E+00
70-30-4	Hexachlorophene	---	---	---	2.0E+01	2.0E+02	---	1.1E+01
1888-71-7	Hexachloropropene	---	---	---	---	---	---	---
591-78-6	2-Hexanone	---	---	---	---	---	---	---
193-39-5	Indeno(1,2,3-cd)pyrene	0.9	---	14	NA	2.6E+00	---	9.2E-02
78-83-1	Isobutyl alcohol	---	---	---	1.1E+04	1.0E+05	---	1.8E+03
465-73-6	Isodrin	---	---	---	---	---	---	---
78-59-1	Isophorone	670	4600	0.5	NA	2.0E+03	---	7.1E+01
120-58-1	Isosafrole	---	---	---	---	---	---	---
143-50-0	Kepone	---	---	---	2.5E-02	1.1E-01	---	3.7E-03
7439-92-1	Lead	400	---	---	NA	4.0E+02	---	4.0E+00
7439-97-6	Mercury (total)	23	10	2	NA	5.1E+02	---	1.1E+01
126-98-7	Methacrylonitrile	---	---	---	2.0E+00	8.1E+00	---	1.0E+00
91-80-5	Methapyrilene	---	---	---	---	---	---	---
72-43-5	Methoxychlor	390	---	160	NA	3.4E+03	40	1.8E+02
74-83-9	Methyl bromide; Bromomethane	110	10	0.2	NA	2.3E+01	---	8.7E+00
74-87-3	Methyl chloride; Chloromethane	---	---	---	1.2E+00	2.6E+00	---	1.5E+00
56-49-5	3-Methylcholanthrene	---	---	---	---	---	---	---
74-95-3	Methylene bromide; Dibromomethane	---	---	---	6.5E+02	6.8E+03	---	3.7E+02
75-09-2	Methylene chloride; Dichloromethane	85	13	0.02	NA	1.8E+01	---	4.3E+00
78-93-3	Methyl ethyl ketone; MEK	---	---	---	7.1E+03	2.7E+04	---	1.9E+03
74-88-4	Methyl iodide; Iodomethane	---	---	---	---	---	---	---
80-62-6	Methyl methacrylate	---	---	---	7.6E+02	2.8E+03	---	4.9E+02
66-27-3	Methyl methanesulfonate	---	---	---	---	---	---	---
91-57-6	2-Methylnaphthalene	---	---	---	---	---	---	---
298-00-0	Methyl parathion; Parathion methyl	---	---	---	1.6E+01	1.7E+02	---	9.1E+00
108-10-1	4-Methyl-2-pentanone; Methyl isobutyl ketone	---	---	---	7.7E+02	2.8E+03	---	1.6E+02

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
91-20-3	Naphthalene	3100	---	84	NA	2.4E+02	---	2.4E+02
130-15-4	1,4-Naphthoquinone	---	---	---	---	---	---	---
134-32-7	1-Naphthylamine	---	---	---	---	---	---	---
91-59-8	2-Naphthylamine	---	---	---	---	---	---	---
7440-02-0	Nickel	1600	13000	130	NA	3.4E+04	---	7.3E+02
88-74-4	o-Nitroaniline	---	---	---	3.9E+00	4.1E+01	---	2.2E+00
99-09-2	m-Nitroaniline	---	---	---	---	---	---	---
100-01-6	p-Nitroaniline	---	---	---	---	---	---	---
98-95-3	Nitrobenzene	39	92	0.1	NA	9.4E+01	---	3.4E+00
88-75-5	o-Nitrophenol	---	---	---	---	---	---	---
100-02-7	p-Nitrophenol	---	---	---	---	---	---	---
56-57-5	4-Nitroquinoline 1-oxide	---	---	---	---	---	---	---
924-16-3	N-Nitrosodi-n-butylamine	---	---	---	2.2E-02	5.5E-02	---	2.0E-03
55-18-5	N-Nitrosodiethylamine	---	---	---	3.0E-03	1.3E-02	---	4.5E-04
62-75-9	N-Nitrosodimethylamine	---	---	---	8.7E-03	3.7E-02	---	1.3E-03
86-30-6	N-Nitrosodiphenylamine	130	---	1	NA	3.9E+02	---	1.4E+01
621-64-7	N-Nitrosodipropylamine; Di-n-	0.09	---	0.00005	NA	2.7E-01	---	9.6E-03
10595-95-6	N-Nitrosomethylethylamine	---	---	---	2.0E-02	8.7E-02	---	3.1E-03
59-89-2	N-Nitrosomorpholine	---	---	---	---	---	---	---
100-75-4	N-Nitrosopiperidine	---	---	---	---	---	---	---
930-55-2	N-Nitrosopyrrolidine	---	---	---	2.1E-01	9.1E-01	---	3.2E-02
99-55-8	5-Nitro-o-toluidine	---	---	---	1.3E+01	5.8E+01	---	2.0E+00
56-38-2	Parathion	---	---	---	3.9E+02	4.1E+03	---	2.2E+02
1336-36-3	Polychlorinated biphenyls; PCBs	1	---	---	NA	3.4E-01	---	8.7E-03
See Note c)	Polychlorinated dibenzo-p-dioxins; PCDDs	---	---	---	---	---	---	---
See Note c)	Polychlorinated dibenzofurans; PCDFs	---	---	---	---	---	---	---
608-93-5	Pentachlorobenzene	---	---	---	5.2E+01	5.5E+02	---	2.9E+01
76-01-7	Pentachloroethane	---	---	---	---	---	---	---
82-68-8	Pentachloronitrobenzene	---	---	---	1.7E+00	7.3E+00	---	2.6E-01
87-86-5	Pentachlorophenol	3	---	0.03	NA	7.9E+00	---	5.6E-01

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
62-44-2	Phenacetin	---	---	---	---	---	---	---
85-01-8	Phenanthrene	---	---	---	---	---	---	---
108-95-2	Phenol	47000	---	100	NA	1.0E+05	---	2.2E+04
106-50-3	p-Phenylenediamine	---	---	---	1.2E+04	1.0E+05	---	6.9E+03
298-02-2	Phorate	---	---	---	1.3E+01	1.4E+02	---	7.3E+00
109-06-8	2-Picoline	---	---	---	---	---	---	---
23950-58-5	Pronamide	---	---	---	4.9E+03	5.1E+04	---	2.7E+03
107-12-0	Propionitrile; Ethyl cyanide	---	---	---	---	---	---	---
129-00-0	Pyrene	2300	---	4200	NA	1.0E+02	---	1.8E+02
110-86-1	Pyridine	---	---	---	6.5E+01	6.8E+02	---	3.7E+01
94-59-7	Safrole	---	---	---	---	---	---	---
7782-49-2	Selenium	390	---	5	NA	8.5E+03	50	1.8E+02
7440-22-4	Silver	390	---	34	NA	8.5E+03	---	1.8E+02
93-72-1	Silvex; 2,4,5-TP	---	---	---	5.2E+02	5.5E+03	50	2.9E+02
100-42-5	Styrene	16000	1500	4	NA	6.8E+02	---	1.6E+03
18496-25-8	Sulfide	---	---	---	---	---	---	---
93-76-5	2,4,5-T; 2,4,5-Trichlorophenoxyacetic acid	---	---	---	6.5E+02	6.8E+03	---	3.7E+02
1746-01-6	2,3,7,8-TCDD; 2,3,7,8-Tetrachlorodibenzo-p-dioxin	---	---	---	3.8E-06	2.4E-05	---	4.5E-07
95-94-3	1,2,4,5-Tetrachlorobenzene	---	---	---	2.0E+01	2.0E+02	---	1.1E+01
630-20-6	1,1,1,2-Tetrachloroethane	---	---	---	2.4E+00	5.4E+00	---	4.3E-01
79-34-5	1,1,2,2-Tetrachloroethane	3	0.6	0.003	NA	1.1E+00	---	5.5E-02
127-18-4	Tetrachloroethylene; Perchloroethylene; Tetrachloroethene	12	11	0.06	NA	1.7E+01	5	1.1E+00
58-90-2	2,3,4,6-Tetrachlorophenol	---	---	---	2.0E+03	2.0E+04	---	1.1E+03
3689-24-5	Tetraethyl dithiopyrophosphate	---	---	---	2.0E+03	2.0E+04	---	1.1E+03
7440-28-0	Thallium (total)	---	---	0.7	5.4E+00	1.2E+02	2	---
7440-31-5	Tin	---	---	---	4.6E+04	1.0E+05	---	2.2E+04
108-88-3	Toluene	16000	650	12	NA	8.8E+02	1000	7.2E+02
95-53-4	o-Toluidine	---	---	---	---	---	---	---

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

CAS No.	Constituent	Soil Screening Level (mg/kg)					GW Screening Level (ug/L)	
					Region 9 PRG			
		Ingestion	Inhalation	Protection of GW [a]	Residential	Industrial	MCL	R9 PRG [b]
8001-35-2	Toxaphene	0.6	89	31	NA	1.7E+00	3	6.1E-02
120-82-1	1,2,4-Trichlorobenzene	780	3200	5	NA	5.5E+03	70	1.9E+02
71-55-6	1,1,1-Trichloroethane;	---	1200	2	NA	3.0E+03	200	7.9E+02
79-00-5	1,1,2-Trichloroethane	11	1	0.02	NA	1.5E+00	5	2.0E-01
79-01-6	Trichloroethylene; Trichloroethene	58	5	0.06	NA	7.0E+00	5	1.6E+00
75-69-4	Trichlorofluoromethane	---	---	---	3.8E+02	1.3E+03	---	1.3E+03
95-95-4	2,4,5-Trichlorophenol	7800	---	270	NA	6.8E+04	---	3.7E+03
88-06-2	2,4,6-Trichlorophenol	58	200	0.2	NA	1.7E+02	---	6.1E+00
96-18-4	1,2,3-Trichloropropane	---	---	---	1.4E-03	3.1E-03	---	1.6E-03
126-68-1	O,O,O-Triethyl phosphorothioate	---	---	---	---	---	---	---
99-35-4	sym-Trinitrobenzene	---	---	---	3.3E+00	3.4E+01	---	1.8E+00
7440-62-2	Vanadium	550	---	6000	NA	1.2E+04	---	2.6E+02
108-05-4	Vinyl acetate	78000	1000	170	NA	2.6E+03	---	4.1E+02
75-01-4	Vinyl chloride	0.3	0.03	0.01	NA	3.5E-02	2	2.0E-02
108-38-3	m-Xylene	160000	420	210	NA	3.2E+02	---	1.4E+03
95-47-6	o-Xylene	160000	410	190	NA	3.2E+02	---	1.4E+03
106-42-3	p-Xylene	160000	460	200	NA	3.2E+02	---	---
1330-20-7	Xylene (total)	160000	410	190	NA	3.2E+02	10000	1.4E+03
7440-66-6	Zinc	23000	---	12000	NA	1.0E+05	---	1.1E+04

a) Migration to groundwater with a dilution attenuation factor of 20. It should be noted that at sites where little or no dilution or attenuation of soil leachate concentrations would be expected between the source and a nearby receptor, a DAF of 20 may not be conservative enough. This situation may apply at sites with shallow water tables, fractured media, karst topography, or source size greater than 30 acres. For such situations, the *Technical Background Document* should be consulted to determine migration to ground water SSLs based on a DAF = 1 (no dilution). Alternatively, a site-specific DAF may be developed using the methodology given in the *Technical Background Document*.

b) Region 9 PRG "Tap Water" value.

c) Note: If the PCDDs and/or PCDFs are suspected as potential constituents of concern, then the analysis must be conducted to identify and quantify the mixture of toxic PCDD/PCDF congeners. The results of congener analysis should be used to calculate a Toxic Equivalent (TEQ) concentration for the mixture. The TEQ concentration should be compared to the RBSLs for 2,3,7,8-TCDD. Please refer to the document: "Estimating Exposure to Dioxin-Like Compounds; Volume II: Properties, Sources, Occurrence and Background Exposures" (EPA/600/6-88/005Cb; June 1994).

d) For this constituent, refer to the values listed under corresponding mixture of isomers.

See text for source references. "---" = value is not currently available. "NA" = value is not applicable for this constituent: refer to the SSL values given in the columns labeled "Ingestion", "Inhalation" and "Protection of GW."

ATTACHMENT E

SOIL SAMPLING STANDARD OPERATING PROCEDURES

SOP 1.0: Soil Sampling

1.0 Purpose

This procedure describes the methods which will be used to establish sampling locations and collect soil samples for laboratory analysis.

2.0 Tools and Materials

The following tools and materials are required to obtain soil samples for off-site laboratory analysis.

Stainless steel trowel or spoons
Stainless steel auger
Stainless steel bowls
Encore sampler
Encore T-handle and extension
Disposable gloves
Sample containers
Sample location flags
Indelible pen

3.0 Sample Collection

Sample locations will be identified as described in Section 2.3 of the Sampling and Analysis Plan (SAP). All sampling equipment used to collect soil samples will be decontaminated as described in section 3.7 of the SAP, and sampling personnel will don clean disposable gloves prior to collecting the samples.

Three categories of soil sampling exist for this project:

1. pre-excavation samples requiring inorganic or SVOC analysis;
2. post-excavation samples requiring inorganic or SVOC analysis;
3. pre and post-excavation samples requiring VOC analysis.

3.1 Pre-excavation samples requiring inorganic or SVOC analysis

These soil samples will be collected from the surface to a depth of 12 inches and will be obtained with a hand auger equipped with a stainless steel bucket.

The auger will be advanced to a depth of 12 inches (usually with repeated advances to reach the required depth), and all of the soil from the auger will be placed into a stainless steel bowl. Any rocks or organic matter in the sample will be discarded after first removing any soil adhering to it. A stainless steel spoon or trowel will then be used to thoroughly mix and homogenize the sample within the bowl. The soil in the bowl will be quartered and equal portions from each quarter, will be obtained to fill the appropriate sample jars.

3.2 Post-excavation samples requiring inorganic or SVOC analysis

The spoon/trowel will be used to obtain a sample to a depth of 6 inches. All of the soil from the hole will be placed into a stainless steel bowl. Any rocks or organic matter in the sample will be discarded after first removing any soil adhering to it. The spoon or trowel will then be used to thoroughly mix and homogenize the sample within the bowl. The soil in the bowl will be quartered and equal portions from each quarter will be obtained to fill the appropriate labeled sample jars. The sample jars will be tagged and placed in a cooler on ice to await shipment.

3.3 Pre and post-excavation samples requiring VOC analysis

These soil samples will be collected from the bottom of the hole made during either pre-or post-excavation sampling activities.

One 5-gram Encore sampler will be snapped into the handle (or handle extension, as necessary) and advanced into undisturbed soil, filling the sampler. While the sampler remains locked in the handle, the cap will be locked onto the sampler. Three Encore samplers will be filled in this manner for every sample. The three samplers will be placed into a labeled sealable bag, tagged, and placed in a cooler on ice to await shipment.

All sample locations will be marked with sample location flags, and the sample identification number will be written in indelible ink on the flag.

4.0 QA/QC Sample Collection

Extra jars for field duplicates and MS/MSDs will be filled in the same manner and at the same time as the corresponding regular sample. Two extra Encore samplers will be obtained for VOC samples that require MS/MSD analysis.

Rinse blanks will be obtained by rinsing the decontaminated sampling equipment with reagent grade water and collecting the water with a funnel directly into the appropriate sample containers. One rinse blank will be collected for every 20 regular samples obtained.

Trip blanks for VOC analysis will accompany every cooler that contains an aqueous sample for VOC analysis. Pre-filled trip blanks will be supplied by the laboratory.

APPENDIX C

QUALITY ASSURANCE PROJECT PLAN

FINAL

QUALITY ASSURANCE PROJECT PLAN FOR SWMU 12/14

MINE FILL A BATTERY SITE CLEANUP AT THE NAVAL SURFACE WARFARE CENTER, CRANE, INDIANA

CONTRACT NO. N68950-96-D-0052
DELIVERY ORDER # FC04
TOLTEST PROJECT NO. 37826.01

FEBRUARY 2001

Submitted to:

OFFICER IN CHARGE OF NAVFAC CONTRACTS
NAVAL SURFACE WARFARE CENTER
BUILDING 2516
300 HIGHWAY 361
CRANE, INDIANA 47522-5082

Submitted by:

TOLTEST, INC.
1915 NORTH 12TH STREET
P.O. BOX 2186
TOLEDO, OHIO 43603
(419) 241-7175

NSWC CRANE

FINAL

QUALITY ASSURANCE PROJECT PLAN

SWMU 12/14

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NAVAL SURFACE WARFARE CENTER CRANE, INDIANA

AUGUST 2000

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(419) 241-7175

PREPARED/APPROVED BY:

TolTest NSWC Facility Manager

Lance Parsons (Date)

APPROVALS:

TolTest QA/QC Representative

Debbie Leighty (Date)

TolTest Program Manager

Ernest C. Enrique, P.E. (Date)

CLIENT ACCEPTANCE:

U. S. Navy Responsible Authority

Brent Robertson (COTR) (Date)

Christine Freeman (ECOTR) (Date)

U.S. EPA Region V

Peter Ramanauskas (Date)

TABLE OF CONTENTS

Page No.

1.0 INTRODUCTION C-1

2.0 PROJECT DESCRIPTION C-2

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY C-3

3.1 Management Responsibilities C-3
3.2 Laboratory Responsibilities C-3
3.2.1 Laboratory Project Manager/Sample Custodian C-3
3.2.2 Laboratory Operations Manager C-4
3.2.3 Laboratory Quality Assurance Officer C-4
3.2.4 Laboratory Technical Staff C-4
3.3 TolTest's Responsibilities C-5
3.1 QA/QC Representative C-5
3.2 Data Reviewer C-5
3.3 Field Staff C-5

4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT OF DATA C-6

4.1 Precision C-6
4.2 Accuracy C-7
4.3 Representativeness C-7
4.4 Comparability C-7
4.5 Completeness C-8
4.6 Level of Quality Control Effort C-8

5.0 SAMPLING LOCATIONS AND PROCEDURES C-11

6.0 CUSTODY PROCEDURES C-12

6.1 Laboratory Custody Procedures C-12
6.2 Final Evidence Files C-12

7.0 CALIBRATION AND STANDARDIZATION PROCEDURES C-13

8.0 ANALYTICAL PROCEDURES C-15

TABLE OF CONTENTS (Continued)

Page No.

9.0 INTERNAL QUALITY CONTROL CHECKS C-17

9.1 Field Quality Control Checks C-17
9.2 Laboratory Quality Control Checks C-17

10.0 DATA REDUCTION, VALIDATION, AND REPORTING C-19

10.1 Data Reduction C-19
10.1.1 Field Data Reduction Procedures C-19
10.1.2 Laboratory Data Reduction Procedures C-19
10.2 Data Validation C-20
10.2.1 Procedures Used to Evaluate Field Data C-20
10.2.2 Procedures to Validate Laboratory Data C-20
10.3 Data Reporting C-21
10.3.1 Field Data Reporting C-21
10.3.2 Laboratory Data Reporting C-22

11.0 PERFORMANCE AND SYSTEM AUDITS C-24

11.1 Field Performance and System Audit C-24
11.2 Laboratory Performance and Systems Audits C-24

12.0 PREVENTIVE MAINTENANCE C-26

12.1 Field Equipment Preventive Maintenance C-26
12.2 Laboratory Instrument Preventive Maintenance C-26

13.0 PRECISION, ACCURACY AND COMPLETENESS C-27

13.1 Precision C-27
13.2 Accuracy C-27
13.3 Completeness C-28

14.0 CORRECTIVE ACTION C-29

14.1 Field Corrective Action C-29
14.2 Laboratory Corrective Action C-30
14.3 Corrective Action during Data Validation and Data Assessment C-31

TABLE OF CONTENTS (Continued)

TABLES

Table 1.0 Summary of Pre-Excavation Sampling and Analysis Program C-9
Table 2.0 SOP Calibration Procedures C-13
Table 3.0 Battery Area Constituent UST C-15
Table 4.0 Soil Area Constituent List C-16

ATTACHMENTS

Attachment A Laboratory Assurance Management Plan
Attachment B Laboratory Operating Procedures with Supplemental SOP Binder
Attachment C Quanterra Quality Control Program
Attachment D Remedial Goals

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) focuses primarily on the analytical methods and the Quality Assurance/Quality Control (QA/QC) procedures that are used to collect and analyze soil samples from the Mine Fill A Battery Site. Issues addressed by this QAPP include the project description, project laboratory organization and responsibilities, analytical procedures, equipment calibration, data reduction, review, reporting, and corrective action.

2.0 PROJECT DESCRIPTION

This project includes furnishing all labor, transportation, supervision, material and equipment, and performing all operations in connection with the excavation, sampling, and disposal of potentially contaminated soil at SWMU 12/14, Mine Fill A Battery Site Cleanup. Since this is an interim measure, this scope of work addresses only the removal of the contamination source and does not address the potential for groundwater contamination.

A further discussion of project description and objectives are presented in Section 1.0 of the Sampling and Analysis Plan (SAP), Appendix B to the Battery Site Work Plan.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

3.1 Management Responsibilities

Project management organization and responsibilities are presented in Section 2.0 of the Work Plan.

3.2 Laboratory Responsibilities

Samples will be shipped to Quanterra at 880 Riverside Parkway, West Sacramento, California, 95605 for analysis. Analytical requirements are listed in Section 2.4 of Appendix B to the Work Plan.

Following is the project laboratory organization and responsibilities of key laboratory personnel. This information is also presented in Quanterra's Quality Assurance Management Plan (QAMP), Attachment A to this plan. Validation of data by TolTest is discussed in section 10.2.2 of this plan.

3.2.1 Laboratory Project Manager/Sample Custodian

The Laboratory Project Manager will report directly to the TolTest Project Manager and will be responsible for the following:

- Ensuring all resources of the laboratory are available on an as-required basis; and
- Providing overview of final analytical reports.
- Receiving and inspecting the incoming sample containers;
- Recording the condition of the incoming sample containers;
- Signing appropriate documents;
- Verifying chain-of-custody and its correctness;
- Notifying the Laboratory Operations Manager of sample receipt and inspection;
- Assigning a unique identification number and customer number, and entering each into the sample receiving log;
- Initiating transfer of the samples to appropriate Laboratory sections; and
- Controlling and monitoring access/storage of samples and extracts.

3.2.2 Laboratory Operations Manager

The Laboratory Operations Manager will report to the Laboratory Project Manager and will be responsible for:

- Coordinating laboratory analyses;
- Supervising in-house chain-of-custody;
- Scheduling sample analyses;
- Overseeing data review;
- Overseeing preparation of analytical reports; and
- Approving final analytical reports prior to submission to the U.S. Navy.

3.2.3 Laboratory Quality Assurance Officer

The Laboratory QA Officer has the overall responsibility for data after it leaves the laboratory. The Laboratory QA officer will be independent of the laboratory but will communicate data issues through the Laboratory Project Manager. In addition, the Laboratory QA officer will:

- Overview laboratory quality assurance;
- Perform internal data validation;
- Overview QA/QC documentation;
- Conduct detailed data review;
- Perform QA audits;
- Determine whether to implement laboratory corrective actions, if required:
- Define appropriate laboratory QA procedures;
- Prepare laboratory Standard Operation procedures; and
- Sign the title page of the QAPP.

3.2.4 Laboratory Technical Staff

The laboratory technical staff will be responsible for sample analysis and identification of corrective actions. The staff will report directly to the Laboratory Operations Manager.

3.3 TolTest's Responsibilities

3.1 QA/QC Representative

The QA/QC Representative will implement and manage the QC program on this project. This individual will remain independent of direct job involvement and day-to-day operations, and will have direct access to corporate executive staff as necessary to resolve any QA dispute. He/she is responsible for auditing the implementation of the QA program in conformance with this plan and TOLTEST'S policies.

3.3.2 Data Reviewer

The TolTest Data Reviewer is responsible for the verification and validation of the analytical laboratory data to determine whether sampling and analysis requirements specified in the SAP and Sections 3 and 4 of Attachment B to the Battery Area Work Plan have been met. In addition, the completeness and correctness of the data reports will be evaluated based on the quality control measures performed by the laboratory. All data will be verified by reviewing the laboratory data and associated documentation following the procedures outlined in Section 10.2.2 of this plan. Upon completion of the data verification, ten percent of the sample data will be validated according to the USEPA Contract Laboratory Program Laboratory Data Validation Functional Guidelines for Evaluating Organic and Inorganic Analysis and/or NEESA 20.2-047B, Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program. Data validation procedures are discussed in Section 10.2.2 of this plan.

3.3.3 Field Staff

It will be the responsibility of TolTest's Environmental Specialist (ES) to implement quality control measures in the field and ensure that the procedures for sample collection, equipment decontamination, sample labeling, sample shipping, and chain of custody documentation are performed according to plan requirements. The ES will ensure that quality control samples (i.e. blanks and duplicates) are obtained at the required intervals.

4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT OF DATA

The overall QA objective is to execute the project in a quality manner. This objective is achieved through implementation of protocols for:

- Field activities, including sample collection and handling
- Chain of custody
- Equipment calibration
- Laboratory analyses
- Data validation
- Data assessment and reporting
- Internal quality control checks
- Audits
- Preventative maintenance
- Corrective action

Sufficient data must be collected to monitor the performance of remedial activities and to confirm attainment of the cleanup goals. The SAP describes the specific sampling and analytical approach to fulfill the objectives. Specific DQOs include precision, accuracy, representativeness, comparability, and completeness.

4.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per 10 analytical samples. Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) and relative standard deviations (RSD) for three or more replicate samples. The equations to be used for precision in this project can be found in Section 13.1 of this plan. Precision control limits are included in Quanterra's standard operating procedures (SOPs), Attachment B to this plan.

4.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation and holding times. Laboratory accuracy is assessed through the analysis of matrix spikes (MS) or standard reference materials (SRM) and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 13.2 of this plan. Accuracy control limits are included in the SOPs.

4.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Work Plan and SAP are followed and that proper sampling techniques are used. Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample-holding times and analyzing and assessing field-duplicated samples.

4.4 Comparability

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Work Plan and SAP are followed and that proper sampling techniques are used. Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

4.5 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount expected to be obtained under normal conditions. Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 13.3 of this plan. Field completeness for this project will be 90% or greater for each area.

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 13.3 of this plan. Laboratory completeness for this project will be greater than 95 percent.

4.6 Level of Quality Control Effort

Field blank, method blank, duplicate, standard reference materials, temperature blanks, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Field blanks (both equipment blanks and trip blanks) consisting of reagent grade water will be submitted to the analytical laboratory to provide the means to assess the quality of the data resulting from the field sampling program. Equipment blank samples are analyzed to check for procedural contamination of the field sampling equipment, which may cause contamination of samples. Trip blank samples are only analyzed for VOCs to determine if samples or containers were contaminated during the collection, packaging or transportation process. Trip blanks are sent in every cooler shipped to the laboratory that contains aqueous samples for VOC analysis.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Duplicate samples are analyzed to check for sampling and analytical reproducibility. The general level of the QC effort will be one field duplicate for approximately every 10 samples.

Matrix spikes (MS) provide information about the effect of the sample matrix on the digestion and measurement methodology. A matrix spike duplicate (MSD) is a second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with the associated sample and MS sample. The results of the MS and MSD are used together to determine the effect of a matrix on the accuracy and precision of the analytical process. Soil MS/MSD samples require two extra EnCore samplers for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix.

Temperature blanks are included in every cooler shipped to the laboratory to check if temperature requirements are being met during shipment.

The estimated number of quality control samples to be collected during pre-excavation sampling activities is listed in Table 1.0. The number of post-excavation samples will not be determined until after excavation activities are complete.

Table 1.0
Summary of Pre-Excavation Sampling and Analysis Program

Sample Matrix	Laboratory Parameters	Excavation Samples	MS/MSD	Duplicates	Equipment Blanks	Trip Blanks	Background
Pre-Excavation Soil Samples – Soil Area	Explosives, App. IX parameters	1	1	1	1 (analysis will be determined by which sample event the blank is obtained)	VOCs (will be sent in cooler with Equipment rinse blank requiring VOC analysis)	4 (analysis to be determined by pre-excavation sampling and analysis)
	MFA Constituents: Explosives, Metals, VOCs	4					
Pre-Excavation Soil Samples – Battery Area	Total metals*, TCLP Metals, Reactivity, Corrosivity, Ignitability	6	1	1			

*Total Metals: Cobalt, Lithium, Manganese, Nickel, Zinc

The number of post-excavation samples will be determined by the size of the excavation areas. Analysis of post-excavation samples will be determined by analysis of pre-excavation sample analysis. The frequency of quality control samples for post-excavation sampling will be the same as used for pre-excavation sampling.

5.0 SAMPLING LOCATIONS AND PROCEDURES

The number of samples to be collected and the sampling locations are described in detail in Section 2.3 of the SAP, Appendix B to the Work Plan. The number of samples is dependent on the size of the areas of suspected contamination. A total of five samples will be obtained from the Soil Area, six samples will be obtained from the Battery Area, and four Background samples will be obtained. Analytical methodologies are discussed in Section 8.0 of this plan.

6.0 CUSTODY PROCEDURES

Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence file. Custody procedures for field sample collection are discussed in Section 4.0 of the SAP.

6.1 Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and log in, sample storage and numbering, tracking during sample preparation and analysis, and storage of data are described in Quanterra's QAMP, Attachment A of this plan. Examples of laboratory chain of custody reports along with instructions for completion are also included.

6.2 Final Evidence Files

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities as described in this plan. TolTest is the custodian of the evidence file and maintains the contents in a limited access area. The contents of this file includes all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews. At the time the final Interim Measures Report is submitted to the USEPA for approval as a final remedy, the evidence file will then be turned over to the NSWC Crane Environmental Protection Department.

7.0 CALIBRATION AND STANDARDIZATION PROCEDURES

The procedures for calibration and standardization can be found in the laboratory SOPs, Attachment B to this plan. The SOPs are listed in Table 2.0 below and the page on which the calibration procedure can be found in its respective SOP is listed.

Table 2.0
SOP Calibration Procedures

Parameter	METHOD	Page #
Metals	6010	15
Explosives	8330	18
Herbicides	8151	13
Pest./PCBs	8082	9
Cyanide	9010	30
Sulfide	9030	9
Ignitability	1010	5
Corrosivity	EPA 150.1	7
Reactive Sulfide	SW846 Sec. 7.3.4.2	7
Reactive Cyanide	SW846 Sec. 7.3.3.2	7
TCLP	1311	13
VOCs	8260/5035	22
SVOCs	8270	14
Mercury	7471	14

Calibration procedures for a specific laboratory instrument will consist of an initial calibration, initial calibration verification and continuing calibration verification. Five point initial calibration curves are utilized as per SOPs in Attachment B. Three point continuing calibration standards or check standards may be used. All calibration must be met in order to proceed with the analysis. In all cases, the initial calibration will be verified using the same or an independently prepared calibration verification solution. Quanterra's Quality Control Program is included as Attachment C to this plan.

The laboratory maintains a sample logbook for each instrument that will contain the following information: instrument identification, serial number, date of calibration, analyst, calibration solutions run and the samples associated with these calibrations.

8.0 ANALYTICAL PROCEDURES

Quanterra will implement the project required SOPs (Attachment B to this plan). These laboratory SOPs for sample preparation, cleanup and analysis are based on U.S. EPA Test Methods for Evaluating Solid Waste (SW-846) Washington, D.C.; Third Edition (September 1986) and revision update III (December 1996). These SOPs contain the latest promulgated information, provide sufficient details, and are specific to this project.

Samples from the battery area will be analyzed for toxic characteristics and metals as listed in Table 3.0 below.

Table 3.0
Battery Area Constituent List

CONSTITUENT	METHOD
Ignitability	1010
Corrosivity	EPA 150.1
Reactive Sulfide	SW846 Sec. 7.3.4.2
Reactive Cyanide	SW846 Sec. 7.3.3.2
RCRA Metals	6010 & 7471
Cobalt	6010
Lithium	6010
Manganese	6010
Nickel	6010
Zinc	6010

The additional metals chosen (over and above the 8 RCRA metals) were based on constituents listed in several Material Data Safety Sheets (MSDSs) for batteries, and Appendix IX metals. Copies of the MSDSs are provided as Attachment 3 to the SAP.

Samples from the soil area will be analyzed for the constituents listed in Table 4 below.

Table 4.0
Soil Area Constituent List

CONSTITUENT	METHOD
1 Sample	
Explosives	8330
Appendix IX list*	various
4 Samples (Mine Fill A constituents)	
Explosives	8330
Metals	6010, 7471
VOCs	8260/5035

* without dioxins or furans

The Appendix IX list can be found as Attachment 2 to the SAP. Analysis for VOCs will conform to SW-846, method 8260/5035 for EnCore samplers. The parameter list for the Soil Area was discussed and agreed upon by the USEPA, the NSWC Crane Environmental Contracting Officer Technical Representative (ECOTR), and TolTest representatives.

Remedial goals, methodologies, and laboratory reporting limits for all analyses are listed as Attachment D to this plan. Remedial goals were taken from the EPA Region 5 Model QAPP App. C (Ecological DQLs for soil) and App. D (RBSSLs). Other industrial risk-based values were published in the *USEPA Region 5 Site-Specific Risk Assessment Memorandum for NSWC Crane* dated February, 1998.

If pre-excavation sampling reveals constituents above the industrial risk based screening levels (RBSLs) presented in Attachment D, or basewide background levels for inorganic constituents, the NSWC Crane ECOTR will determine the appropriate cleanup goals for detected constituents above RBSLs at the site. Based on the selection of industrial or residential remediation goals, the affected soils will be removed to meet these goals. Post-excavation sampling will confirm that the cleanup goals have been met and it will be determined if these areas will be included for investigation in the RFI for the Mine Fill A SWMU.

For constituents detected and for which the detection limit is not low enough to achieve the risk-based level (or a risk-based level does not exist), excavation will proceed to the detection limit and the area will be included for further study under the RFI/Risk Assessment for Mine Fill A.

If pre-excavation sampling does not reveal constituent concentrations above RBSLs or basewide background for inorganics, no excavation will occur.

Samples will be analyzed in the laboratory on standard turnaround time of one to three weeks depending on analytical methodology used. Site restoration not will begin until the Navy has approved the analytical data from the confirmation samples.

9.0 INTERNAL QUALITY CONTROL CHECKS

9.1 Field Quality Control Checks

The QC information for the field is stated in Section 4 of this QAPP. Collection of the samples will be according to the applicable procedures in Section 5 of this QAPP.

9.2 Laboratory Quality Control Checks

Quanterra has a QC program it uses to ensure the reliability and validity of the analysis done at the laboratory. All analytical procedures are documented in writing as SOPs and each SOP includes a QC section that addresses the minimum QC requirements for the procedure. The internal quality control checks might differ slightly for each individual procedure but overall the QC requirements include the following:

Equipment blanks - analyzed to check for procedural contamination of sampling equipment, which may cause contamination of the confirmation samples.

Trip blanks - analyzed to check for contamination of VOC samples during shipment to the laboratory.

Method blanks - Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures.

Reagent/preparation blanks (applicable to inorganic analysis) - Reagent blanks consist only of the reagents used to prepare samples, and they are analyzed the same way as the test samples. They identify and/or correct the signals produced by the reagents or impurities in the reagents.

Instrument blanks - Instrument blanks (system blanks) are solvent or reagent blanks that measure interference or contamination from an analytical instrument.

Matrix spikes/matrix spike duplicates - Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology of organics analysis. Duplicate samples are analyzed to check for sampling and analytical reproducibility.

Analytical spikes (Method Spike, Matrix Spike) - measure the normal level bias due to matrix effects and analytical method errors, including laboratory contamination, calibration errors, etc. Method spikes consist of solvent or reagent blanks spiked with the analytes of interest. Matrix spikes consist an aliquot of the sample spiked with analytes of interest.

Field duplicates - Field duplicate samples are analyzed to check for sampling and analytical reproducibility of field samples.

Laboratory duplicates - Laboratory duplicate samples are analyzed to check for sampling and analytical reproducibility of laboratory samples.

Laboratory control standards (QC check standards) - require certified standards, generally supplied by an outside source. They are used to measure normal level bias originating from procedural or operator errors or contamination from laboratory sources.

All data obtained will be properly recorded. The data package can allow the recipient to reconstruct QC information and compare it with QC criteria. Any samples analyzed in nonconformance with the QC criteria will be reanalyzed by the laboratory, if sufficient volume is available. It is expected that sufficient volumes/weights of samples will be collected to allow for reanalysis when necessary.

10.0 DATA REDUCTION, VALIDATION, AND REPORTING

All data generated through field activities or by the laboratory operation will be reduced and validated before reporting. The laboratory will distribute no data until it has been subjected to these procedures summarized in subsections below:

10.1 Data Reduction

10.1.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared with those implemented in the laboratory setting. Since field instrumentation is not anticipated to be utilized on this project, there will be no further need for assuring that field data has been reduced properly through formulas or interpretation of raw data printouts.

10.1.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be followed according to the following protocol: All raw analytical data will be recorded in numerically identified laboratory notebooks. These notebooks will be issued only by the Laboratory QA Manager. Data are recorded in this notebook along with other pertinent information, such as the sample identification number and the sample label number. Other details will also be recorded in the Laboratory notebook, such as the analytical method used, name of analyst, the date of analysis, matrixes sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook will be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the Laboratory QA Manager takes place before final data reporting. The Laboratory QA Manager maintains records of notebook entry inspections.

Quality control data (e.g., laboratory duplicates, matrix spikes, and matrix spike duplicates) will be compared with the method acceptance criteria. Data considered acceptable will be entered into the laboratory computer system. Data summaries will be sent to the Laboratory QA Manager for review. Once approved, data are logged into the project database format. Unacceptable data will be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell

outside acceptance limits, and any other anomalous conditions encountered during sample analysis. After the Laboratory QA Manager approves these data, they are considered ready for third party data validation.

10.2 Data Validation

Data validation procedures will be performed for both field and laboratory operations as described below.

10.2.1 Procedures Used to Evaluate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of field logbooks, by field crewmembers. This task will be the responsibility of the field QCR.

10.2.2 Procedures to Validate Laboratory Data

USEPA Contract Laboratory Program Laboratory Data Validation Functional Guidelines for Evaluating Organic and Inorganic Analysis will be used as the primary guide to validate laboratory data. Parameters not included in the Functional Guidelines will be validated following the criteria outlined in Section 7 of NEESA 20.2-047B, Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program. Qualifiers will be applied to the data based on results of the validation effort.

Data verification is a preliminary review to ensure that samples are labeled, preserved, shipped, maintained, and analyzed in accordance with the data quality objectives, sample analysis plan, and the work plan. The Data Reviewer will verify all data by reviewing the analytical laboratory data and associated documentation, including chain-of-custody records, sample preservation records, analytical holding times, sample data and reporting requirements. Data will be reviewed for timeliness of the report, chain-of-custody compliance, accurate sample identifications, compliance with extraction and holding times, and quality of analytical data. The Data Reviewer will conduct a systematic review and evaluation of the quality control performed by the laboratory for compliance with established criteria and to address the completeness and correctness of the data report based on the standard and sample spikes, sample duplicates, method blanks, and

surrogate spikes. All method detection limits and results of initial and continuing calibration will be reviewed and evaluated.

Checks for entirety of deliverables will be administered on all data reports to decide whether all deliverables specified in this QAPP are present. At a minimum, deliverables will include sample chain-of-custody forms, analytical results, quality control summaries, and supporting raw data from instrument printouts. The reviewer will determine whether all required items are present and request copies of missing deliverables.

Following the data verification, ten percent of the data from each data package will be validated following the accepted criteria defined in the references listed in the first paragraph of this section. The Data Reviewer will identify any out-of-control data points and data omissions and interact with the laboratory to correct any data deficiencies. The Project Manager will decide whether or not to repeat sample collection and analyses based on the extent of the deficiencies and their importance in the overall context of the project.

All data generated for the Mine Fill A Battery Site will be computerized in a format organized to facilitate data review and evaluation. The computerized data set will include the data flags provided by Quanterra according to the Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (February 1994) and Inorganic Analyses (February 1994), and additional comments of the Data Reviewer. The laboratory-provided data flags will include such items as: 1) estimated concentration due to poor spike recovery, and 2) concentration of chemical also found in laboratory blanks.

The following qualification flags are used by the laboratory:

E = Estimated concentration, use when compounds exceed the upper range of the calibration curve. (Preliminary Report only).
D = Result was obtained from the analysis of a dilution.
J = Estimated result, used when the result is less than the sample quantitation limit but greater than zero.
B = Used when an analyte is found in the associated blank as well as in the sample.
NC = Recovery and/or RPD were not calculated.
MSB = Recovery and RPD were not calculated because sample amount was greater than four time the spike amount.

a = Spike analyte recovery is outside stated control limits.
N = Spike analyte recovery is outside stated control limits.
* = Relative Percent Difference (RPD) is outside stated control limits.

The Data Reviewer will also evaluate completeness of the data package. Completeness checks will be administered on all data to decide whether deliverables specified in the QAPP are present. At a minimum, deliverables will include sample chain-of-custody forms, analytical results, QC summaries, and supporting raw data from instrument printouts if requested. The reviewer will determine whether all required items are present and request copies of missing deliverables.

10.3 Data Reporting

Data reporting procedures will be carried out for field and laboratory operations as indicated below:

10.3.1 Field Data Reporting

Field data reporting will be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field activities.

10.3.2 Laboratory Data Reporting

The task of reporting laboratory data begins after the validation activity has been concluded. The Laboratory QA Manager must perform a final review of the report summaries and case narratives to decide whether the report meets project requirements. A Data Validation Report will be submitted for the validated data. Besides the record of chain-of-custody, the report format will consist of the following:

A. Case Narrative:
- Date of issuance
- Laboratory analysis performed
- Any deviations from intended analytical strategy
- Laboratory batch number
- Numbers of samples and respective matrices

- Quality control procedures used and references to the acceptance criteria
- Laboratory report contents
- Project name and number
- Condition of samples "as-received"
- Discussion of whether or not sample holding times were met
- Discussion of technical problems or-other observations that may have created analytical difficulties
- Discussion of any laboratory quality control checks which failed to meet project criteria
- Discussion of any corrective actions taken.
- Signature of the Laboratory QA Manager

B. Chemistry Data Package:

- Case narrative for each analyzed batch of samples
- Summary page indicating dates of analyses for samples and laboratory quality control checks
- Cross referencing of laboratory samples to project sample identification numbers
- Data qualifiers to be used should be adequately described
- Sample preparation and analyses for samples
- Sample results
- Raw data for sample results and laboratory quality control samples, including chromatograms
- Results of (dated) initial and continuing calibration checks
- Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, and surrogates

A Case Narrative and Chemistry Data Package will be organized per analytical type. In each grouping, acceptance criteria will be listed, including a listing of all samples effected and their data qualifiers, and reasoning why this particular data is qualified. The Chemistry Data Package is immediately made available for review.

11.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analyses are performed according to the procedures established in the Work Plan and SAP. The audits of field and laboratory activities include two independent parts: internal and external audits.

11.1 Field Performance and System Audit

Internal audits of field activities including sampling and field measurements may be conducted by the TolTest QA Officer. These audits verify that all established procedures are being followed. Internal field audits may be conducted at the beginning of the site sample collection activities.

The audits include examination of field sampling records, field instrument operating records, sample collection, handling and packaging according to the established procedures, maintenance of QC procedures, chain-of-custody, etc. Follow up audits may be conducted to correct deficiencies, and to verify that QC procedures are maintained throughout the project. The audits involve review of field measurement records, instrumentation calibration records, and sample documentation.

External field audits may be conducted by the U.S. Navy, IEPA or USEPA Region 5. External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the U.S. Navy or USEPA Region 5. External field audits will be conducted according to the field activity information presented in the QAPP.

11.2 Laboratory Performance and Systems Audits

The internal laboratory audit will be conducted by Quanterra's QA staff. The internal Laboratory system audits will be done annually while the internal Laboratory performance audits will be conducted quarterly.

The internal laboratory system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. Quanterra 's QA staff will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

External laboratory audits may be conducted by the NSWC, U.S. Navy and IDEM or USEPA Region 5. External laboratory audits may be conducted at least once before the initiation of the sampling and analysis activities. These audits will be announced and are at the discretion of the NSWC, IDEM or USEPA Region 5. External laboratory audits will include, but not limited to, review of the laboratory analytical procedures, the laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

12.0 PREVENTIVE MAINTENANCE

12.1 Field Equipment Preventive Maintenance

There is no field instrumentation scheduled for use on this project.

12.2 Laboratory Instrument Preventive Maintenance

As part of their QA/QC Program, a routine preventive maintenance program is conducted by Quanterra to reduce the occurrence of instrument failure and other system malfunctions. The laboratory has an internal group to do routine scheduled maintenance, and to repair or to coordinate with the vendor for the repair of all instruments. All laboratory instruments are maintained according to manufacturers' specifications and the requirements of the specific method employed. This maintenance is carried out on a regular, scheduled basis, and is documented in the laboratory instrument service logbook for each instrument. Emergency repair or scheduled manufacture's maintenance is provided under a repair and maintenance contract with factory representatives.

13.0 PRECISION, ACCURACY AND COMPLETENESS

13.1 Precision

Spiked samples are prepared by choosing a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and then spiking each aliquot with a known amount of analyte. The duplicate samples will be then included in the analytical sample set. The splitting of the sample allows the analyst to decide the precision of the preparation and analytical techniques associated with the duplicate sample. The relative percent difference (RPD) between the spike and duplicate spike will be calculated according to the following formula:

$$RPD = \frac{\text{Amount in Spike 1 - Amount in Spike 2}}{0.5\,(\text{Amount in Spike 1 + Amount in Spike 2})} \times 100$$

13.2 Accuracy

To assure the accuracy of the analytical procedures, an environmental sample will be randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte or analytes to be evaluated. In general, a sample spike will be included in every set of 20 samples tested on each instrument. The spike sample will be then analyzed. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared with the reported value of the same analyte in the unspiked sample determines the percent recovery. Daily control charts will be plotted for each commonly analyzed compound and kept on instrument-specific, matrix-specific, and analyte-specific bases. The percent recovery for a spiked sample is calculated according to the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample - Amount in Sample}}{\text{Known Amount Added}} \times 100$$

13.3 Completeness

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{(number of valid measurements)}}{\text{(number of measurements planned)}} \times 100$$

14.0 CORRECTIVE ACTION

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of quality control performance that can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All corrective action proposed and implemented will be documented in the regular quality assurance reports to management. Corrective action should only be carried out after approval by the Project Manager, or his designee. If immediate corrective action is required, approvals secured by telephone from the Project Manager should be documented in an additional memorandum.

For non-compliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem will be responsible for notifying the Project Manager, who in turn will notify the NSWC. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPP or SAP will be identified and corrected according to the QAPP. Corrective actions will be carried out and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped.

14.1 Field Corrective Action

Corrective action in the field can be needed when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the SAP, etc.), or sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. TolTest's Environmental Specialist will be responsible for reporting all suspected technical or QA non-conformances or suspected deficiencies to the ECOTR. If appropriate, no additional work that is dependent on the nonconforming activity will be attempted until the corrective actions are completed.

14.2 Laboratory Corrective Action

Corrective action in the laboratory may occur before, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, potentially high concentration samples, may be identified during sample log-in or just before analysis. Following consultation with laboratory analysts and section leaders, approving the start-up of corrective action may be necessary for the Laboratory Quality Assurance Officer. The submitted SOPs specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, an additional sample extract cleanup, automatic re-injection/re-analysis when certain quality control criteria are not met, etc.

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken may be dependent on the analysis and the event. Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain target analytes above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- Unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits;
- From the results of performance evaluation samples; or
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department.

Corrective actions are done before releasing data from the laboratory. The corrective actions will be documented in Quanterra's laboratory's corrective action log (signed by analyst, section leader and quality control coordinator), and the narrative data report sent from the laboratory to the TolTest Data Reviewer. If corrective action does not rectify the situation, the laboratory will contact TolTest's Data Reviewer who will notify TolTest's Project Manager.

14.3 Corrective Action during Data Validation and Data Assessment

The need for corrective action may be identified during either data validation or data assessment. Potential types of corrective action may include re-sampling by the field team or re-injection/re-analysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team or whether the data to be collected is necessary to meet the required quality assurance. It is the responsibility of TolTest's Project Manager for approving the start-up of corrective action. If corrective action is required, the Project Manager will notify the ECOTR who would then be responsible for notifying the U.S. EPA.

ATTACHMENT A

LABORATORY QUALITY ASSURANCE MANAGEMENT PLAN

Quanterra®

Quality Assurance Management Plan

Approved by:

Mark A. Matthews
Chief Operating Officer

Chris M. Lee
Vice President of Operations Services

Jack R. Hall
Corporate Director of Quality Assurance

Revision 3
November 2, 1998

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Preface

The purpose of the Quanterra® Quality Assurance Management Plan (QAMP) is to provide internal quality assurance (QA) guidance to Quanterra® operating units. This guidance allows Quanterra® to operate under a standardized, rigorous quality management system (QMS) and ensures that our clients are consistently provided with data that are of known and documented quality and are legally defensible. The QAMP outlines the purpose, policies, organization, responsibilities, and operations related to ensuring high quality performance in all Quanterra® activities. The QAMP also fulfills the requirement of our clients and of government programs to document our QMS.

The QAMP contains many references to other essential Quanterra® quality documents. These quality documents, including the QAMP, Policy Documents, and Standard Operating Procedures (SOPs), both corporate and laboratory-specific, help ensure the quality of our products and intertwine to produce a strong QMS within Quanterra®. This system is the foundation that provides our operations with guidance and ensures consistently-produced quality deliverables. The project-specific requirements delineated in project plans may supersede the general quality requirements described in this manual.

The document is designed to follow the basic outline required for a quality management plan as described by the United States Environmental Protection Agency (EPA). Table 2.3-1 cross-references the narrative sections of the QAMP to the appropriate sections of the EPA document and other nationally recognized quality standards. This revision reflects specific changes to meet the Quality Standard Requirements of the National Environmental Accreditation Conference (NELAC) dated July, 1998.

There are two basic types of information included in the QAMP. General information is applicable to all Quanterra® operating units. Operation-specific information, provided in the Facility-Specific Appendix, describes the quality control (QC) requirements that apply only to a specific laboratory. The Facility-Specific Appendix includes information such as method detection limits (MDLs), performance evaluation (PE) studies, and laboratory SOPs that cannot be standardized throughout the Quanterra® laboratory network due to client-specific, laboratory-specific, or instrument-specific nature.

Forward

QAMP Revision 3 Implementation:

Quality Assurance Managers are to distribute the document and conduct training on the changes to the QAMP within 45 days of the issuance date. Implementation of the changes to the QAMP shall be completed within 60 days of the issuance date.

Table of Contents

Page

Table of Contents
Title and Signature Page i
Copyright Statement ii
Preface iii
Forward iv
Table of Contents v
List of Figures xi
List of Tables xii
List of Appendices xiv
Acronyms and Initialisms xv

1.0 Management Commitment and Organization 1
1.1 Vision Statement 1
1.2 Statement of Management Commitment to Quality 1
1.3 Organizational Structure and Relationships 2
1.4 Quality Organization 4
1.4.1 President and Chief Executive Officer (CEO) 4
1.4.2 Chief Operating Officer 4
1.4.3 Vice President of Operations Services 4
1.4.4 Corporate Director of Quality Assurance 4
1.4.5 Assistant Quality Assurance Director 5
1.4.6 Quality Assurance (QA) Manager 5
1.4.7 Regional General Manager 6
1 4.8 Laboratory Manager 6
1.4.9 Operations Manager 7
1.4.10 Laboratory Technical Manager 7
1.4.11 Systems Manager 7
1 4.12 Customer Service Managers (CSMs) 8
1.4.13 Project Manager 8
1.4.14 Group Leader or Team Leader 8
1.4.15 Analyst 9
1.4.16 Sample Custodian 9
1.4.17 Report Production Staff 9
1.5 General Responsibilities 9
1.6 Waste, Fraud, and Abuse 9

2.0 Quality System and Description 11
2.1 Quality Management System 11
2.2 Quality Assurance 11

Table of Contents (continued)

Page

2.0 Quality System and Description (continued)
2.3 Quality Documents 12
2.3.1 Quality Assurance Management Plan 13
2.3.2 Quality Policy Documents 13
2.3.3 Standard Operating Procedures 13
2.3.4 Quality Assurance Project or Program Plans 13
2.3.4.1 Quality Assurance Summary (QAS) 14
2.3.5 Other Documents 14

3.0 Associate Qualification and Training 15
3.1 Associate Qualifications 15
3.2 Orientation and Technical Training of Laboratory Staff 15
3.2.1 Quality Orientation 16
3.2.2 Quality Training 16
3.2.3 Health and Safety Orientation and Training 17
3.2.4 QA Manager Training 17
3.3 Training Files 17
3.3.1 Associate Resumes 17
3.3.2 Individual Training Records for the Areas of Quality, Health and Safety, and Technical Proficiency Monitoring 17
3.3.3 Training Records for Professional Development 18
3.3.4 Training Records for Regulatory/Compliance Information 18

4.0 Procurement of Items and Services 19
4.1 Selection of Vendors 19
4.2 Procurement of Quality-Related Items 20
4.2.1 Role of Corporate Director of Contracts 20
4.2.2 Role of Quanterra® Purchasing 21
4.2.3 Procurement Procedures 21
4.2.4 Evaluation of QRIs 22
4.2.5 Special Requirements for Standard Reference Materials 22
4.3 Procurement of Subcontract Laboratory Services 23
4.4 Vendor Partnerships 24

5.0 Documentation and Records Keeping System 25
5.1 Quality Documents and Records 25
5.2 Document Review and Revision 25
5.3 Document Control and Distribution 25

Table of Contents (continued)

Page

5.0 Documentation and Records Keeping System (continued)
5.4 Effective Dates and Document History 26
5.5 Records Management 27
5.5.1 Quality Records 27
5.5.2 Project Records 28
5.5.3 Electronic Data 28
5.6 Storage, Retention and Disposal of Records 29
5.7 Data Confidentiality 29

6.0 Computer Hardware and Software 31
6.1 Use of Hardware 31
6.2 Security 31
6.3 Use of Software 31
6.3.1 Industry Standard Software 32
6.3.2 Quanterra® -Developed Software 32
6.3.3 Control of Software Changes 32
6.3.4 Software Revalidation 32
6.4 Documentation 33
6.5 Computer Viruses 33

7.0 Planning 35
7.1 Data Collection Process 35
7.2 Organizational Responsibilities 35
7.3 Determination of Project QC Requirements 37
7.4 Communication of Project-Specific Requirements 38
7.5 Customer Service Teams 38
7.6 Contingency Planning 38

8.0 Work Processes and Operations 43
8.1 Standard Operating Procedures 43
8.2 Analytical Methods 43
8.3 Data Quality Objectives 44
8.3.1 Precision and Accuracy 45
8.3.2 Completeness 45
8.3.3 Representitiveness 46
8.3.4 Comparability 46
8.3.5 Method Detection Limits 46
8.3.6 Instrument Detection Limits 46

Table of Contents (continued)

Page

8.0 Work Processes and Operations (continued)

8.3.7 Reporting Limits 47
8.4 Quality Control Samples 48
8.4.1 Field QC Samples 48
8.4.1.1 Trip Blank 49
8.4.1.2 Rinsate Blank 49
8.4.1.3 Field Blank 49
8.4.1.4 Field Duplicate 49
8.4.1.5 Field Matrix Spike 49
8.4.1.6 Collocated Samples 49
8.4.1.7 Split Sample 50
8.4.2 Laboratory QC Samples 50
8.4.2.1 Quality Control (QC) Batch 51
8.4.2.2 Method Blank 51
8.4.2.3 Instrument/Calibration Blank 51
8.4.2.4 Laboratory Control Sample 52
8.4.2.5 Matrix Spike 52
8.4.2.6 Matrix Spike Duplicate 52
8.4.2.7 Sample Duplicate 53
8.4.2.8 Surrogates 53
8.4.2.9 Analytical Spike 53
8.4.2.10 Interference Check Sample 53
8.4.2.11 Internal Standards 53
8.4.2.12 Radiological QC Samples 54
8.5 Data Collection Operations 54
8.5.1 Field Collection and Shipment 54
8.5.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times 55
8.5.2.1 Sample Containers 55
8.5.2.2 Shipping Containers 57
8.5.2.3 Sample Preservatives 57
8.5.2.4 Sample Holding Times 57
8.5.3 Sample Handling 58
8.5.3.1 Sample Receipt 58
8.5.3.2 Sample Log-in 59
8.5.3.3 Sample Storage 59
8.5.3.4 Internal Sample Chain-of-Custody and Interlaboratory Transfers 62

Table of Contents (continued)

Page

8.0 Work Processes and Operations (continued)

8.5.3.5 Sample Disposal and Return Chain-of-Custody 63
8.5.4 Calibration Procedures and Criteria 63
8.5.4.1 Physical Reference Standards 64
8.5.4.2 Chemical Reference Standards and Reagents 64
8.5.4.3 Standard Verification 65
8.5.4.4 Periodic Calibration 65
8.5.4.5 Operational Calibration 66
8.5.4.6 Calibration Failure 66
8.5.4.7 Calibration Records 66
8.6 Quality Assessment 67
8.6.1 Data Quality Assessment 67
8.6.2 Statistical Evaluation of Data 67
8.7 Data Recording Procedures 68
8.8 Data Reduction and Verification Procedures 68
8.8.1 Data Reduction and Initial Verification 68
8.8.2 Data Verification 71
8.8.3 Completeness Verification 72
8.9 Data Reporting 72
8.9.1 Data Reports 72
8.9.2 Verbal Results 73
8.9.3 Reporting Analytical Results 73
8.10 Data Validation 74
8.11 Preventive Maintenance and Service 74
8.11.1 Analytical Instrumentation and Equipment 74
8.11.2 Facilities 75
8.11.3 Frequency of Maintenance 75
8.12 Other Requirements 76
8.12.1 Water 76
8.12.2 Compressed Air and Gases 76
8.12.3 Glassware Preparation 76
8.12.4 Chemical Storage 76
8.12.5 Waste Disposal 77
8.12.6 Facility Security 77

Table of Contents (continued)

Page

9.0 Quality Assessment and Response 79
9.1 Nonconformance and Corrective Action 79
9.1.1 Nonconformance 79
9.1.2 Corrective Action 79
9.1.3 Responsibilities 80
9.1.4 Nonconformance Memo 80
9.2 Audits 80
9.2.1 Performance Audits 81
9.2.2 Systems Audits 81
9.2.2.1 Independent Internal Systems Audits or Evaluations 81
9.2.2.2 External Systems Audits 83
9.2.3 Data Audits 83
9.2.4 Spot Assessments 83
9.3 Client Inquiries and Complaints 84
9.4 Quality Reports to Management 84
9.5 Management Process Review 85
9.6 Management Review of the QMS 85

10.0 Quality Improvement 87
10.1 Standardization of Procedures 87
10.2 Continuous Quality Improvement 87
10.3 Benchmarking 88
10.4 Quality Assessment 88
10.5 Understanding the Clients' Needs 88
10.6 Quality Measures and Standards 88

List of Figures

Figure	Title	Page
1.3-1	Quanterra® Organizational Chart	3
7.1-1	Data Collection Process Flow Diagram	36
7.4-1	Example Quanterra® Quality Assurance Summary	39-40
8.5-1	Example Quanterra® Chain-of-Custody Form	56
8.5-2	Example Quanterra® Condition Upon Receipt Anomaly Report (CUR)	60-61
8.8-1	Data Reduction, Verification, and Reporting	69

List of Tables

Table No.	Title	Page
2.3-1	Quanterra® Quality Assurance Management Plan Requirements Matrix	5
2.3-2	Cross-Reference of QAMP Sections Addressing NELAC Quality Manual Requirements	9
4.2-1	List of Quanterra® Quality-Related Items that Require Evaluation Prior to Use	12
5.1-1	Quanterra® Quality Documents and Required Approval	13
5.2-1	Quanterra® Quality Document Review Requirements	14
8.4-1	Field Quality Control Samples	15
8.4-2	Laboratory Quality Control Samples	16
8.4-3	Laboratory Performance Quality Control Samples	17
8.4-4	Matrix Specific Quality Control Samples	17
8.4-5	Inorganic Laboratory Quality Control Samples	18
8.4-6	Organic Laboratory Quality Control Samples	67
8.4-7	USEPA Contract Laboratory Program Statement of Work Quality Control Samples	96
8.5-1	Inorganic Sample Containers, Preservatives, and Holding Times	110
8.5-2	Organic Sample Containers, Preservatives, and Holding Times	122
8.5-3	Radiological Sample Containers, Preservatives, and Holding Times	136
8.5-4	Sample Containers, Preservatives, and Holding Times for USEPA Contract Laboratory Program Statement of Work	139
8.5-5	Sample Containers, Preservatives, and Holding Times for TCLP	141
8.5-6	Periodic Equipment Calibrations	142
8.5-7	Summary of Inorganic Method Calibrations	143
8.5-8	Summary of Organic Method Calibrations	159
8.5-9	Summary of USEPA Contract Laboratory Program Statement of Work Method Calibrations	169
8.6-1	Precision and Accuracy Measurements	173
8.11-1	Instrument Maintenance Schedule - Ion Chromatograph	175
8.11-2	Instrument Maintenance Schedule - LACHAT Auto Analyzer	175
8.11-3	Instrument Maintenance Schedule - Total Organic Halide Analyzer	176
8.11-4	Instrument Maintenance Schedule - High Pressure Liquid Chromatograph	176
8.11-5	Instrument Maintenance Schedule - Flame Atomic Absorption Spectroscopy	177
8.11-6	Instrument Maintenance Schedule - Inductively Coupled Argon Plasma/ Mass Spectrometry (ICAP/MS)	177
8.11-7	Instrument Maintenance Schedule - ICP	178
8.11-8	Instrument Maintenance Schedule - Graphite Furnace Atomic Absorption	179
8.11-9	Instrument Maintenance Schedule - Cold Vapor Atomic Absorption (Leeman PS 200)	179

List of Tables (continued)

Table No.	Title	Page
8.11-10	Instrument Maintenance Schedule - Cold Vapor Atomic Absorption (PE 5000)	180
8.11-11	Instrument Maintenance Schedule - Gas Chromatograph	181
8.11-12	Instrument Maintenance Schedule - Mass Spectrometer	183
8.11-13	Instrument Maintenance Schedule - TRAACS 800 Auto Analyzer	184
8.11-14	Instrument Maintenance Schedule - Sonicator	184
8.11-15	Instrument Maintenance Schedule - Analytical/Top Loading Balances	184
8.11-16	Instrument Maintenance Schedule - Refrigerators/Walk-in Coolers	184
8.11-17	Instrument Maintenance Schedule - Ovens	185
8.11-18	Instrument Maintenance Schedule - Specific Digital Ion Analyzer	185
8.11-19	Instrument Maintenance Schedule - Turbidimeter	185
8.11-20	Instrument Maintenance Schedule - Dissolved Oxygen Meter	185
8.11-21	Instrument Maintenance Schedule - Conductance Meter	186
8.11-22	Instrument Maintenance Schedule - Chemical Oxygen Demand (COD) Reactor	186
8.11-23	Instrument Maintenance Schedule - Spectrophotometer	186
8.11-24	Instrument Maintenance Schedule - pH Meter	187
8.11-25	Instrument Maintenance Schedule - Fourier Transform Infrared Spectrometry	187
8.11-26	Instrument Maintenance Schedule - Radiological Analysis Equipment	188
8.11-27	Instrument Maintenance Schedule - Total Organic Carbon Analyzer (OI 7000)	189
8.11-28	Instrument Maintenance Schedule - APCI/ESI LC/MS/MS	190
8.11-29	Instrument Maintenance Schedule - Digestion Block	191
8.11-30	Instrument Maintenance Schedule - Flash Point Tester	191

List of Appendices

Appendix A — Corporate Key Personnel List and Quanterra® Operations Organizational Chart

Appendix B — Addresses of Quanterra® Locations

Appendix C — Facility-Specific Appendix: ***This appendix contains facility-specific quality-related information and requirements for*** Quanterra® ***laboratories. These laboratories are located in the following cities:***

- Anchorage, Alaska
- Austin, Texas
- City of Industry, California
- Denver, Colorado
- Knoxville, Tennessee
- North Canton, Ohio
- Pittsburgh, Pennsylvania
- Richland, Washington
- Sacramento, California
- Santa Ana, California
- St. Louis, Missouri
- Tampa, Florida

Each laboratory section in this appendix contains information specific to that laboratory only and contains the following basic outline:

Section	Contents
0	Table of Contents
1	Organizational Chart
2	Instrument List
3	Standard Operating Procedures List
4	Analytical Methods
5	MDLs, RLs, and CRDLs
6	Performance Evaluation Studies
7	Additional Operation-Specific Information

Acronyms and Initialisms

A2LA	American Associatio for Laboratory Accreditation
AA	Atomic Absorption
ANSI	American National Standards Institute
AR/COC	Analysis Request/Chain-of-Custody
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BLK	Blank
BOD	Biochemical Oxygen Demand
CCC	Calibration Check Compound
CEO	Chief Executive Officer
CF	Calibration Factor
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
CLP	Contract Laboratory Program
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act (Superfund)
COC	Chain-of-Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CSM	Customer Service Manager
CSRM	Certified Standard Reference Material
CST	Customer Service Team
CUR	Condition Upon Receipt
CV	Coefficient of Variation
CVAA	Cold Vapor Atomic Absorption (Spectroscopy)
DFTPP	Decafluorotriphenylphosphine
DOC	Dissolved Organic Carbon
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective

Acronyms and Initialisms (continued)

EH&S	Environmental Health and Safety
EPA	(U. S.) Environmental Protection Agency
FAS	Field Analytical Services
FLAA	Flame Atomic Absorption (Spectroscopy)
FTIR	Fourier Transform Infrared (Spectrometry)
GC	Gas Chromatograph(y)
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption (Spectroscopy)
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
ICAP	Inductively Coupled Argon Plasma (Spectroscopy)
ICAP/MS	Inductively Coupled Argon Plasma/Mass Spectrometry
ICS	Interference Check Sample
IDL	Instrument Detection Limit
IR	Infrared (Spectroscopy)
IS	Information Systems
IS	Internal Standard
ISO	International Organization for Standardization
IT	Information Technology
KRI	Key Result Indicator
LAN	Local Area Network
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LRGC	Low Resolution Gas Chromatography
LRMS	Low Resolution Mass Spectrometry
LWL	Lower Warning Limit
MBAS	Methylene Blue Active Substance

Acronyms and Initialisms (continued)

MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NELAC	National Environmental Laboratory Accreditation Conference
NCM	Nonconformance Memo
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards Technology
NMOC	Non-Methane Organic Compounds
NPDES	National Pollutant Discharge Elimination System
NRC	Nuclear Regulatory Commission
NRM	National Reference Material
PAH	Polynuclear Aromatic Hydrocarbons (or PNA)
PC	Personal Computer
PCB	Polychlorinated Biphenyls
PDS	Post Digestion Spike
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
PM	Project Manager
PQL	Practical Quantitation Limit
PSRL	Project-Specific Reporting Limit
PUF	Polyurethane Foam
QA	Quality Assurance
QAMP	Quality Assurance Management Plan
QAPP	Quality Assurance Project Plan or Quality Assurance Program Plan
QAS	Quality Assurance Summary
QC	Quality Control
QMS	Quality Management System
QuantIMS	Quanterra® Laboratory Information Management System

Acronyms and Initialisms (continued)

QRI	Quality-Related Item
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFP	Request for Proposal
RFQ	Request for Quote
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compounds
SPLP	Synthetic Precipitation Leaching Procedure
SRL	Standard Reporting Limit
SRM	Standard Reference Material
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TOX	Total Organic Halides
UCL	Upper Control Limit
UPS	Uninterruptable Power Supply
USEPA	United States Environmental Protection Agency
UWL	Upper Warning Limit
VOA	Volatile Organic Analysis
VOST	Volatile Organic Sampling Train
WAN	Wide Area Network
WS	Water Supply
WP	Water Pollution

1.0 Management Commitment and Organization

1.1 Vision Statement

Quanterra®'s vision is to be a world-class, profitable analytical services company providing high-value, compliant, innovative problem solutions and state-of-the-art products at a competitive price wherever they are required.

1.2 Statement of Management Commitment to Quality

Quanterra®'s management is committed to providing quality services that meet the requirements of our clients and satisfy applicable regulatory requirements. Management is dedicated to providing an environment that encourages the achievement of excellence, demands integrity in all aspects of its operations, and requires active participation of all associates and vendors in meeting its quality goals.

A comprehensive Quality Management System (QMS) has been developed to ensure that Quanterra®'s clients receive high-quality analytical and environmental services that are timely, reliable, and meet their intended purpose in a cost-effective manner. The QMS provides the organizational structure that ensures quality in its work processes, products, and services. The Quanterra® QMS is described in this Quanterra® Quality Assurance Management Plan (QAMP) and applies to all technical, business, and administrative functions at Quanterra®. The principles and practices described in this QAMP apply to all Quanterra® associates at every level and are fundamental to the services we provide and to the way we do business.

The Quanterra® QAMP provides the foundation for planning, implementing, and assessing the Quanterra® QMS. It is an overall statement of quality policy as well as a plan used to implement quality programs throughout the company. Each business function of the organization shall put in place plans, policies, and procedures that will meet the requirements of the QAMP. The QAMP provides guidance to Quanterra®'s associates in fulfilling their responsibilities and serves as a statement to clients, agencies, and associates of Quanterra®'s commitment to quality. The QAMP by definition documents the policies, elements of, procedures, objectives and commitment to accepted laboratory practices.

Implementation of the QAMP is the responsibility of all Quanterra® associates. Management at every level has the responsibility and authority to lead the development and implementation of a

structured management system that supports the quality programs. Management must ensure that the principles and practices of the quality program are followed and implemented at all levels.

1.3 Organizational Structure and Relationships

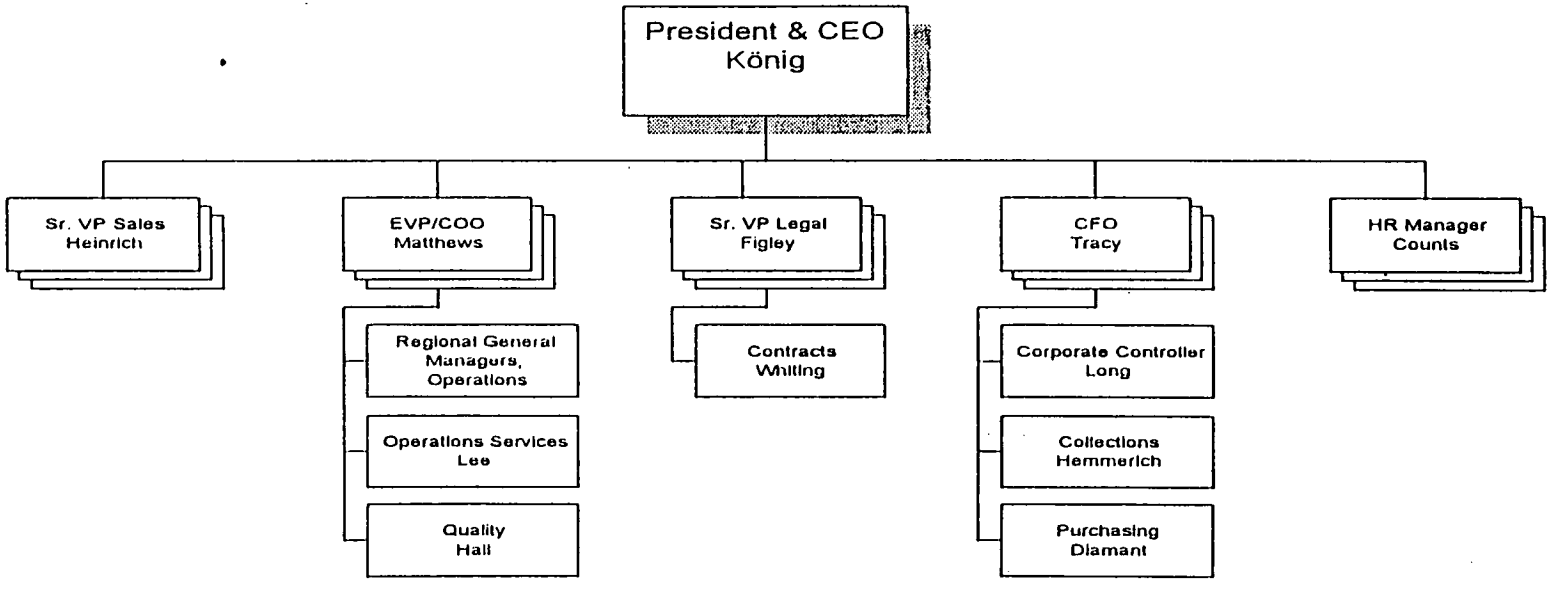
Quanterra® is incorporated in the state of Delaware with corporate headquarters located in Englewood, Colorado. The organizational structure for Quanterra® is presented in Figure 1.3-1. Laboratory locations are given in the List of Appendices in the Table of Contents. A list of key corporate personnel and an organizational chart for Quanterra® operations including Corporate Quality Assurance are given in Appendix A. The organizational structure for each laboratory is presented in the Facility-Specific Appendix C. The responsibilities and authorities of the members of the organization, as they relate to quality management, are outlined in Section 1.4.

At some Quanterra® laboratories, positions identified in this section may not exist due to the laboratory size or other factors. In these cases, the responsibilities and authorities described here are assigned to other positions by the Laboratory Manager or next senior level of management as appropriate.

Each Quanterra® laboratory has day-to-day independent operational authority that is overseen by the various corporate functions (Senior Management, Health and Safety, Technology, Quality Assurance, Finance and Human Resources). The laboratory has, therefore, the operational staff supported by local management, namely, a QA manager, safety coordinator, technical manager, human resources, controller, reporting personnel and administrative support. This team is directed by the laboratory manager to meet daily workload commitments with a degree of autonomy. The team participates in the planning and execution of projects as defined in Section 7.0. These support functions have a level of responsibility to the Corporate level staff and management. Quality Assurance is an example of a function which is independent from the laboratory's day-to-day operations. Even though a key member on the laboratory team, the QAM has direct access to the Corporate Director of Quality Assurance (DQA) on all quality issues and their proper reporting. All QA candidate selection, hiring and disciplinary actions are approved by the Corporate DQA. This approach ensures independence of judgement and integrity without the influence of financial and scheduling pressures.

FIGURE 1.3-1

QUANTERRA ORGANIZATIONAL CHART



as of 5/13/98

1.4 Quality Organization

The achievement of quality in all activities that pertain to their organizational/technical function is the responsibility of each Quanterra® associate. Quality-related responsibilities within the organization provide for the implementation of the QAMP and for completion of quality control (QC) activities. The following sections describe these activities for key Quanterra® positions. The quality-related responsibilities may be reassigned by assigning the activities among different individuals or enhanced by adding activities, but they must not be eliminated. If an operation does not have the titled position outlined below, the responsibilities under that position go to next upward position. The accountability is retained by the person delegating the activity or by the person assigned the activity by the next senior level of management.

1.4.1 President and Chief Executive Officer (CEO)

- Reports directly to the Board of Directors
- Responsible for overall corporate policy and strategy for quality
- Responsible for maintaining effective quality management
- Provides the resources to implement the Quanterra® QAMP.

1.4.2 Chief Operating Officer

- Reports directly to the President and CEO
- Approves the Quanterra® QAMP
- Responsible for implementation of an effective QMS
- Implements improvements in operations and quality programs
- Provides feedback regarding customer satisfaction.

1.4.3 Vice President of Operations Services

- Reports directly to the Chief Operating Officer
- Approves the Quanterra® QAMP and Quality Policy Documents
- Recommends improvements in operations and quality programs
- Oversees evaluation of new technology and required quality control

1.4.4 Corporate Director of Quality Assurance

- Reports directly to the Vice President of Operations Services and Chief Operating Officer on all quality issues
- Reviews and approves changes in laboratory quality assurance (QA) staff

- Maintains a strong line of communication to and provides guidance to the QA Managers

- Approves the Quanterra® QAMP, the Facility Appendices to the QAMP, Quality Policy Documents, and Corporate Standard Operating Procedures (SOP)

- Serves as a technical consultant and resource on quality issues to ensure uniform excellence in quality and regulatory analytical compliance at all Quanterra® operations

- Responsible for assessing, maintaining, and improving the Quanterra® QAMP implementation

- Supervises and provides guidance to corporate QA staff

- Suspends processing when significant quality requirements are not met

- Oversees independent systems and performance audits of Quanterra® laboratories to identify areas where improvement is needed to comply with the QAMP

- Responsible for reporting all matters of quality assurance to the President and senior management.

- Issues Corporate Quality Assurance Directives as required to clarify policy

- Duties completed by Assistant QA Director in absence of the Director

1.4.5 Assistant Quality Assurance (QA) Director

- Reports directly to the Corporate Director of Quality Assurance

- Serves as a back-up to the Corporate Director of Quality Assurance in their absence

- Completes special projects related to corporate QA issues

1.4.6 Quality Assurance (QA) Manager

- Reports directly to the Laboratory Manager and indirectly for all QA matters to the Corporate Director of Quality Assurance

- Approves the Facility-Specific Appendix to the Quanterra® QAMP

- Approves operation-specific SOPs

- Responsible for assessing and maintaining the QAMP implementation within the facility operations

- Responsible for ensuring and improving quality within facility operations

- Recommends resolutions for ongoing or recurrent nonconformances within the laboratory

- Supervises and provides guidance and training to laboratory QA staff

- Responsible for issuing "stop work orders" in analytical areas when significant quality requirements are not met

- Assists in maintaining regulatory analytical compliance
- Serves as the in-house client representative on all project inquiries involving data quality issues
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs QA assessments
- Issues QA directives, policy or local QA SOPs to meet the Corporate requirements or to clarify such requirements for their facility.
- Tracks and closes external and internal findings of QA audits
- Reviews and approves corrective action plans for nonconformances, trends nonconformances to detect systematic problems, and initiates additional corrective actions as needed.
- Assists in the preparation of and approves Quality Assurance Project Plans (QAPPs)
- Serves as a QA officer for significant projects.
- Coordinates laboratory certification and accreditation programs
- Maintains controlled quality documents
- Prepares a monthly quality report to management
- Responsible for approving reference data and changes on LIMS.

1.4.7 Regional General Manager

- Reports directly to the Chief Operating Officer
- Responsible for implementing and adherence to the Quanterra® QAMP in their operating units and approves the QAMP Facility-Specific Appendices for those units.
- Assigns specific responsibilities within operational units to resolve quality-related problems within their region
- Maintains a dialogue with the Chief Operating Officer, regional QAMs and the Corporate Director of Quality Assurance
- Assesses the effectiveness of the Quanterra® QMS within their region.

1.4.8 Laboratory Manager

- Reports directly to the Regional General Manager
- Responsible for implementing and adherence to the Quanterra® QAMP and all corporate policies and procedures within the laboratory
- Approves the Facility-Specific Appendix to the Quanterra® QAMP and Facility-Specific SOPs
- Annually assesses the effectiveness of the QAMP within the operation

- Maintains adequate staffing documented on organization charts
- Responsible for implementing internal/external audit findings corrective actions

1.4.9 Operations Manager

- Reports directly to the Laboratory Manager
- In Operations where this positions does not exist, these responsibilities lie with Group (Area) Leader or Team Leader.
- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs
- Works with the Systems Manager and Group/Team Leaders to assure the requirements of projects are met in a timely manner
- Responsible for meeting quality requirements.

1.4.10 Laboratory Technical Manager

- Reports directly to Laboratory Manager
- Responsible for coordinating development of SOPs
- Performs technical training in area(s) of expertise
- Overall, responsible for a defined technical area(s) of the laboratory. In a small laboratory, these responsibilities may be shared with another position. Other laboratories may have one or more technical managers
- Interfaces with management on technical needs and solving day-to-day technical issues
- Determines qualification required for technical positions and evaluates job candidates against those requirements
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work.
- Certifies the qualification of laboratory personnel.

1.4.11 Systems Manager

- Reports directly to the Laboratory Manager
- Supervises daily activities of the Project Management, and/or Sample Control, and/or Administrative, and/or Report Production Groups
- In some laboratories the CSM may have all or some of the responsibilities of the Systems Manager.

- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner
- Responsible for meeting quality requirements.

1.4.12 Customer Service Managers (CSMs)

- Reports directly to the Laboratory Manager
- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs
- Brings together resources necessary to ensure customer satisfaction.

1.4.13 Project Manager

- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff
- Prepares Quality Assurance Summary (QAS) or equivalent summary form and communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Reviews project data packages for completeness and compliance to client needs
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Conducts project reviews to assess the laboratory's performance in meeting customer requirements
- Prepares reissue requests for project data
- Responsible for meeting quality requirements.

1.4.14 Group (Area) Leader or Team Leader

- Reports directly to either the Systems Manager, or Operations Manager or Laboratory Manager depending on specific laboratory organization.
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations

- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for meeting quality requirements.

1.4.15 Analyst

- Performs analytical methods and data recording in accordance with documented procedures
- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures
- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures
- Responsible for meeting quality requirements.

1.4.16 Sample Custodian

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports anomalies associated with condition-upon-receipt of samples to the Project Manager
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal
- Responsible for meeting quality requirements.

1.4.17 Report Production Staff

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Responsible for meeting quality requirements.

1.5 General Responsibilities

It is the responsibility of each Quanterra® associate to perform their job-related duties in compliance with all Quanterra® corporate and operation-specific SOPs and policies applicable to their position.

1.6 Waste, Fraud, and Abuse

Quanterra®, through its ethics policy (Corporate Policy Number LEG-001) and

ethics training, and the Quanterra® Compliance Program (Corporate Policy Number LEG-009) requires and encourages all associates to report any activity that may be considered wasteful or fraudulent. Any incidents reported are subject to a complete investigation per Quanterra® Corporate Policy Number LEG-005, "Internal Investigation". Associates involved in such activities are subject to immediate dismissal.

Substance abuse is unacceptable behavior for Quanterra® associates and abuses are subject to Human Resources disciplinary policies. Prior to hiring, all Quanterra® associates undergo drug screening and are subject to random screening on an annual basis. The Quanterra® procedures on substance abuse meet the federal contracting guidelines.

2.0 Quality System and Description

Quanterra® has defined Quality as meeting the requirements of our clients, both internal and external. The QMS provides the structure to achieve the total quality management goals necessary to obtain world class standards of performance and quality in all areas.

2.1 Quality Management System

The purpose of the QMS is to ensure the quality of products and services. The QMS is a structured management system of principles, objectives, policies, responsibilities, and implementation plans at the organizational and project-specific levels. At the organizational level, the QMS provides the framework within which project-specific planning, implementation, and performance assessment may occur. The QAMP documents the QMS and describes both the organizational and project-specific principles, goals, controls, and tools of the QMS. The QMS is described in detail in this QAMP, Quality Policy Documents, and SOPs.

The QMS steering committee is comprised of the Quanterra® President and CEO and his staff. This committee establishes the Quanterra® Vision and its strategic plan and provides leadership and support for the achievement of all quality goals and objectives. It is the responsibility of all Quanterra® directors and managers to implement the QMS elements by setting goals and objectives which lead to the achievement of the Quanterra® Vision.

2.2 Quality Assurance

Quality Assurance (QA) is defined as a system of activities which ensures a process, product, or service that meets the needs and expectations of the customer. QA is an integral part of Quanterra® 's QMS.

The organizational and project-specific systems of the Quanterra® QMS, discussed in Section 2.1, are used to define QA goals. Controls at the organizational level regulate activities that support common or standardized functions such as associate qualifications and training, document control, and material procurement. Controls at the project level regulate the definition and implementation of customer requirements to produce the desired type and quality of product. Some specific examples of quality controls are:

- Measuring lab and instrument performance on a daily basis to ensure that the measurement systems are in statistical control

- Demonstrating lab capability through data quality assessments which document the overall qualification of the laboratory to perform environmental analyses

- Utilizing SOPs to ensure uniformity and compliance in the measurement process
- Providing controlled flexibility in routine methodology to meet project-specific sample and data requirements
- Monitoring operational performance of the laboratory on a routine basis and providing corrective action if needed
- Recognizing and promptly correcting any factors which adversely affect quality
- Maintaining complete records of sample receipt, laboratory analysis, data evaluation and reporting, and sample disposal.

2.3 *Quality Documents*

The QMS is defined by a series of documents which are described in Sections 2.3.1 through 2.3.5. The review and control of these documents are described in Section 5.0.

Following is a list of documents used to develop Quanterra®'s QAMP. The requirements of several of these documents are cross-referenced with the content of the QAMP in Table 2.3-1. A cross-reference of the QAMP and Quanterra® documents to NELAC Quality Systems Standards is presented in Table 2.3-2.

- Draft Interim Final EPA Requirements for Quality Management Plans, U.S. Environmental Protection Agency, EPA QA/R-2, August 1994
- EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 Final, August 1997
- Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, American Society for Quality Control, Energy and Environmental Quality Division, Environmental Issues Group, ANSI/ASQC E4-1994 (Formerly EQA-1), January 1994
- Quality Assurance Program Requirements for Nuclear Facilities, The American Society of Mechanical Engineers, ASME NQA-1-1989 edition
- Quality Assurance, Office of Nuclear Energy & Office of Environment, Safety, and Health, United States Department of Energy, DOE ORDER 5700.6C, August 1991
- Title 10 Code of Federal Regulations, Part 830.120 Quality Assurance Requirements
- Implementation Guide for Use with Independent and Management Assessment Requirements of 10CFR, Part 830.120 and DOE 5700.67, DOE G414.1-1, August, 1996
- Implementation Guide for Use with 10CFR, Part 830.120, Quality Assurance, 6830 - Rev. 0, April 15, 1994
- Performance Criteria for Radiobioassay, ANSI N 13.30, September 1989
- Measurement Quality Assurance for Radioassay Laboratories, ANSI N 42.2, Revised May 21, 1992, Revision 10A

- Quality Management and Quality System Elements for Laboratories - Guidelines, American National Standard, American Society for Quality Control, ANSI/ASQC Q2-1991

- International Standard ISO/IEC Guide 25-1990

- Interim Standard National Environmental Laboratory Accreditation Conference Draft Standards January 12, 1998 and Standards dated July 2, 1998

2.3.1 Quality Assurance Management Plan

This Quanterra® Quality Assurance Management Plan (QAMP), along with Quality Policy Documents and SOPs, provides the criteria and specifications for the generation of environmental analytical data. The QAMP provides QC criteria for standard procedures and facility-specific instrumentation as well as method detection limit (MDL) information.

2.3.2 Quality Policy Documents

Quality Policy Documents provide further detail to the QAMP. They describe the requirements for a specific program on a corporate-wide level. Quality Policy Documents use the concepts and requirements contained in the QAMP and provide sufficient detail so that corporate or facility-specific SOPs can be developed.

2.3.3 Standard Operating Procedures

Standard Operating Procedures (SOPs) describe step-by-step instructions for performing a method or activity. In addition, there are SOPs which relate to other support services performed in the company. In general, SOPs will be corporate or operation specific. Corporate SOPs specify procedures that are standard across Quanterra®. Operation-specific SOPs detail procedures that pertain to a specific facility operation only. SOPs specify procedures, methods, corrective action requirements, documentation, review, and verification requirements. SOP format and document control are described in Quanterra® Policy Number QA-001, "Standard Operating Procedures." SOPs that are performed by each Quanterra® operating unit are listed in Section 3 of the Facility Appendix to this QAMP. SOPs are living documents and may supersede some requirements in this document until the QAMP update every two years.

2.3.4 Quality Assurance Project or Program Plans (QAPPs)

Regulations and contracts may contain QA requirements which are different from Quanterra® 's QAMP. To address unique or project requirements, Quality Assurance Project Plans (QAPP) may be prepared and implemented. The requirements documented in a QAPP take precedence over the Quanterra® QAMP for that project.

Similarly, some regulatory programs such as Florida Department of Environmental Protection (FDEP) and Florida Department of Health (FDH) require QA Plans specific to their program. The FDEP and FDH require that each laboratory certified in the state submit and maintain a Comprehensive QA Plan. Many state certifying agencies have reciprocal certification agreements with Florida. In these situations, the QA Plan submitted for regulatory programs takes precedence over the Quanterra® QAMP.

If requested and approved by the client, project-specific requirements may be less stringent than the Quanterra® quality program. Typical specifications contained in a QAPP or documentation include:

- New or modified testing methods
- Unique QC logic
- Special requirements for equipment use and maintenance
- Special handling due to safety considerations
- Project-specific detection and reporting limits
- Project-specific accuracy and precision limits or the statistical treatment of data
- Additional or unique documentation or records management requirements.

2.3.4.1 *Quality Assurance Summary*

Quality Assurance Summaries (QAS) or equivalent are used to distill client-specific requirements typically documented in project QA plans onto a concise format. The summary describes for each project the required quality control samples, batching schemes, flagging conventions, deliverables, or other special client requests that may differ from routine laboratory operations. The QAS or equivalent is disseminated to laboratory operations by the Project Manager to document client-specific requirements. The QAS may be used alone or in conjunction with the project-specific QA plans.

2.3.5 *Other Documents*

Other documents which can affect the quality program may include the Chemical Hygiene Plan (CHP), the Quanterra® Compliance Program , memos, guidance documents, work instructions, and periodic management assessment reports. These documents may further define or guide the implementation of quality standards at Quanterra® but shall not conflict with the QAMP or diminish the effectiveness of the QMS.

3.0 Associate Qualification and Training

All activities performed by Quanterra® shall be accomplished by qualified associates. The following definitions are relevant to the discussion of associate qualification and training presented in this section:

- Qualification - The characteristics or abilities gained through education, training, or experience that enable an individual to perform a required task.
- Orientation - The act or process of acquainting individuals with an existing situation, environment, or condition.
- Training - In-depth instruction to develop proficiency in the application of requirements, methods, and procedures. Instruction may be internal or external classroom sessions, courses, or on-the-job training.

Employee orientation and training must be performed in compliance with Quanterra® Corporate SOP Number CORP-QA-0013, "Employee Orientation and Training".

3.1 Associate Qualifications

Each operating unit shall have job descriptions for all positions. These job descriptions must specify the minimum qualifications for education and experience, knowledge, and skills which are necessary to perform at a satisfactory level. An associate's performance shall be compared with the requirements of his/her job description at least annually, in conjunction with the associate's annual performance review. Due to their length and detailed nature, job descriptions for all employees are provided in a separate Human Resource (HR) Manual available at each laboratory.

Quanterra® expects the necessary knowledge, experience, and skills to be demonstrated by formal academic training. Qualifications of professional associates shall be documented by resumes which include academic credentials, employment history, experience, and professional registrations. A copy of the resume will be placed in the associate's training file or may be maintained in electronic format.

3.2 Orientation and Technical Training of Laboratory Staff

Associates receive internal, external, formal, and informal training. Training is performed to maintain and develop proficiency, and to promote improvement. Training is performed by individuals knowledgeable in the subject matter.

Quanterra® associates are qualified based on the experience and training documented in the individual's training file, and are assigned duties within their experience and training. Each new associate shall receive orientation and training in quality and health and safety. Each new associate shall be supervised in their assigned duties by their supervisor or designee. The authorization to perform independently shall be documented in the training files and must be approved by the Technical Manager or designee. In addition, training for associates may include professional, managerial, communication, and interpersonal skills as appropriate. On-going or periodic assessments will be performed to determine training needs and effectiveness of instruction.

3.2.1 Quality Orientation

Each newly hired Quanterra® associate receives a Quality orientation. The QA Manager or designee shall conduct this orientation within two weeks of the associate's report-to-work date. This orientation will be documented in the associate's training file. The QA Manager shall review the following topics (at a minimum) with the new associate as they apply to individual's assigned responsibilities:

- Quanterra® Quality Policy and applicable documents including the Quanterra® QAMP
- Quanterra® policies on ensuring data integrity, meeting client requirements, and ethics
- Identification and documentation of nonconformance and corrective action procedures
- Proper data recording practices
- Key elements of Quanterra® Quality Control Program Policy (No. QA-003).

The QA orientation or on-going training shall be documented using a written exam, a checklist, or a preprinted form. This documentation is required to demonstrate an understanding of the Quality orientation and to determine if any areas covered require further training.

3.2.2 Quality Training

Continued training in the mission and goals of the QAMP shall be provided at least once per year. Formal training sessions are conducted and documented by the QA Manager or designee. The training program shall address relevant regulatory requirements, basic QC practices, responsibilities of the technical and QA staff, and the reporting of nonconformances.

In addition, each Quanterra® associate shall become familiar with the operating unit's quality programs by reading the relevant

sections of the Quanterra® QAMP, policies, and SOPs pertinent to his/her position.

3.2.3 Health and Safety Orientation and Training

Each newly hired Quanterra® associate, contract worker, or working visitor is required to go through health and safety orientation and training as per the laboratory Chemical Hygiene Plan (CHP). The orientation must be performed as soon as possible after the associate's report-to-work date and before chemicals are handled. Quanterra® associates and contract workers shall be given comprehensive health and safety training within ninety days of the start-to-work date. Documentation is maintained in the associate's training file. A detailed description of this training is also provided in the CHP.

3.2.4 QA Manager Training

All QA Managers shall receive training so that they are proficient in the requirements of the Quanterra® QAMP. Continued proficiency of QA Managers shall be maintained through active participation in QA audits and the preparation and review of QA documents.

3.3 Training Files

Each active Quanterra® associate has an individual training file maintained by the QA Manager or designee. The types of training documents included in the training file are as follows:

- Associate's resume (if not maintained electronically)
- Quality assurance and quality control
- Health and Safety
- Technical proficiency
- Professional Development
- Regulatory/Compliance.

Information is filed in the training file as training is received. Not all associates will have training records for all areas depending upon their job function or tenure with the company.

3.3.1 Associate Resumes

A copy of the associate's current resume will be placed in the associate's training file or maintained in electronic format. Qualifications of associates, as documented on resumes, include academic credentials, employment history, experience, and professional memberships and registrations.

3.3.2 Individual Training Records for the Areas of Quality, Health and Safety, and Technical Proficiency Monitoring

Training of each associate shall be summarized and documented on training

forms or in a data base. These include documentation of participation in training, one-on-one training, on-the-job training initial and ongoing proficiency, or participation in classes and other presentations, and any other formal training sessions, either internal or external. Examples of some of the forms used to document this training are provided in the Corporate SOP, "Employee Orientation and Training", SOP Number CORP-QA-0013. Initial or ongoing technical proficiency training records shall include documentation of the ability to perform sample preparation or analysis using internally prepared laboratory control samples and/or externally available blind standard reference materials per the most current version of the method or SOP. For further details on initial or ongoing technical training refer to "Employee Orientation and Training", SOP Number CORP-QA-0013. In addition, if any tests are given as part of the training in each of these areas, the results are filed in the individual's training file. Technical proficiency of analysts must be approved by the Technical Manager or designee.

3.3.3 *Training Records for Professional Development*

This category includes documentation of all courses taken relating to an individual's professional development. Examples of courses include Conflict Resolution, Management/Supervision, Time Management, Conducting Effective Meetings, and Interviewing Skills.

3.3.4 *Training Records for Regulatory/Compliance Information*

This category provides for documentation of training on topics required by law (with the exception of safety documented previously). Examples of this category are Sexual Harassment, Drug-Free Workplace, and Quanterra® ethics.

4.0 Procurement of Items and Services

This section defines the Quanterra® requirements for the procurement of items and services. Controlling the quality of items and services procured by Quanterra® will help us meet the needs of our customers. The Quanterra® procurement program requires:

- Assurance that purchased items and services meet Quanterra®-established requirements and perform as expected
- Definitions and descriptions of the documentation levels required for the applicable technical and administrative requirements
- Evaluation and qualification of vendors.

4.1 Selection of Vendors

Prospective vendors are selected based upon criteria appropriate to the materials or services provided. For national vendors and contracts, the vendor is selected by the Corporate Director of Contracts through either a competitive proposal/bid process, strategic business alliance or negotiated vendor partnership. Potential vendors are required to complete a vendor acceptance application and are evaluated on the following criteria as appropriate:

- The vendor's history of providing identical or similar products that perform satisfactorily in actual use
- The vendor's service record and ability to provide a complete product line and commensurate service
- The vendor's ability to administer inventory at Quanterra® facilities through a fully developed inventory management system that will ensure correct stocking levels as well as shelf-life tracking
- Software systems that will integrate with Quanterra® systems
- Objective evaluation of the vendor's current quality records supported by documentation
- Ability of the vendor to provide service agreements for instruments that meet Quanterra® specifications
- Evaluation of the vendor's business strategy and the ability of that strategy to complement Quanterra®'s quality program
- Results of audits by Quanterra® of the vendor's technical and quality capability.

A Quanterra® quality representative shall determine the appropriate level of evaluation criteria for the item or service being purchased. Vendors that provide test and measuring equipment, solvents, standards, instrument-related service contracts, or subcontracted laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items. For the procurement of testing and measuring equipment it is recognized that the

environment in which the measurement system is placed may have a bearing on its performance. Therefore, in lieu of other supplier qualification activities, the quality representative shall ensure that an acceptance testing plan is in place to ensure that the measurement system is able to meet specifications in the intended operating environment.

4.2 Procurement of Quality-Related Items

The quality of instruments, equipment, standards, reagents, solvents, other chemicals, gases, water, and laboratory containers used in analyses must be known so that their effect upon analytical results can be defined. Quality-related items (QRIs) are items that are used in the operational unit that must meet a minimum quality requirement. All QRIs purchased by Quanterra® operating units shall be evaluated to ensure that they meet the requirements and specifications established by Quanterra®. These requirements and specifications include, but are not limited to, client contracts, project-specific QA requirements, data quality objectives (DQOs) and analytical method requirements, and defined technical specifications.

Quality specifications shall be included or referenced in the purchasing documents for the procurement of the applicable items. Reference to an approved model, lot number or chemical grade is sufficient. If items which may affect laboratory quality are requested from non-preapproved vendors, quality approval must be obtained prior to placing the order.

When ordering QRIs, a system shall be put in place to verify the quality of the item received. Each laboratory shall assign individuals responsible for material procurement and control. Duties include:

- Specifying, in purchase orders or requisitions, suitable grades of materials (grade shall be defined by the responsible manager and detailed in SOPs)
- Verifying upon receipt that materials meet requirements (see section 4.2.3) and that, as applicable, material certificates are provided and maintained
- Identifying and storing materials
- Verifying that material storage is properly maintained, and removing materials from use when shelf life has expired.

4.2.1 Role of Corporate Director of Contracts

The Corporate Director of Contracts supports the laboratory by:

- Maintaining, issuing, and negotiating contracts
- Identifying potential vendors and subcontractors

- Identifying vendors for unique or scarce materials
- Preparing and communicating corporate policies regarding purchasing and procurement
- Developing and implementing with QA a review of the purchasing program as it pertains to procurement of QRIs.

In order to enhance the quality and consistency of the product within the laboratory network, the Director of Contracts shall pursue national contracts for laboratory supplies, standards, and instruments of known quality and proven reliability.

4.2.2 *Role of Quanterra® Purchasing*

Quanterra® Purchasing supports the laboratory by:

- Identifying vendors for unique or scarce materials
- Maintaining a system to facilitate purchases made by each facility
- Maintaining lists of available items through catalogs or contract agreements with approved vendors
- Preparing and communicating corporate policies regarding purchasing and procurement.

4.2.3 *Procurement Procedures*

The specifications for standards, chemical reagents, solvents, gases, water, and other QRIs shall be documented in SOPs. In addition, each laboratory must have SOPs that cover the following:

- Checking purity of standards, reagents, reagent grade water, and other chemicals as appropriate versus intended use
- Preparation, storage and expiration of standards, reagents, and other chemicals as appropriate
- Requirements for lab containers (e.g., volumetric glassware).

Corporate SOPs will be developed for these processes where appropriate. Operation-specific SOPs shall be in place if a corporate SOP does not exist.

Corrective actions for failure of an item to meet required specifications are as follows:

- Review current supplies and eliminate from use; this must include communication to the Quanterra® laboratory network and corporate purchasing to avoid additional problems in other facilities
- Return to vendor
- Evaluate a new lot or alternate supplier
- Evaluate the impact on product or process.

The Corporate Director of Purchasing or the Corporate Director of Quality Assurance shall be notified promptly of any quality problems with national vendors.

4.2.4 Evaluation of QRIs

Most QRIs will be evaluated as a function of the standard analytical process. This involves the analysis of method blanks, as well as other QC samples as described in Section 8.0. For some QRIs, small variability in the item may significantly affect analytical results. For these QRIs the analysis and evaluation must take place prior to using the item. The Corporate SOP CORP-QA-0001; "Quality Testing of Solvents, Acids, and Reagents (QRI Program)"; documents the testing procedures and acceptance criteria. A list of QRIs that require testing prior to use is listed in Table 4.2-1. If any QRI is determined during routine use to be the source of quality problems, or has demonstrated variability that affects analytical results, the QRI may be added to the list in Table 4.2-1. Facility QA Managers must forward such requests to the Corporate Director of Quality Assurance, the Director of Purchasing, and Corporate Technology.

For items that are used regularly by Quanterra® facilities where no unique requirements or specifications are required, the items may be purchased off-the-shelf. These items are ordered from the supplier on the basis specifications set forth in the supplier's published product description. Off-the-shelf items include general laboratory supplies such as glassware, filter paper, pipettes, and chromatography columns. These items are evaluated as a function of the standard analytical process. An off-the-shelf item can be added to the list in Table 4-2.1 when it is determined that evaluation prior to use is required. Requests to add items to this list are made to the facility QA Manager.

Evaluation of instruments purchased shall be conducted according to the acceptance testing plan as established in the procurement documents. Acceptance criteria may inclu instrument reliability, sensitivity, stability, accuracy, and ability to interface with existing computer systems and networks.

4.2.5 Special Requirements for Standard Reference Materials

For all standard reference materials (SRMs), Quanterra® will use materials of known quality for the intended use. Where available, SRMs will be traceable to the National Institute of Standards Technology (NIST), the American Association for Laboratory Accreditation (A2LA), or to an equivalent source. If the traceability is not commercially accessible, the best available standard for the material or

isotope shall be used. Certificates for Certified Standard Reference Materials (CSRMs) shall be procured from the supplier. Documentation received with each standard shall include the following information as appropriate:

- Traceability to an approved source (where available) or other certificate of analysis
- Radionuclide identification with activity and error
- Reference Material Certificate
- Certification Report that will include pertinent information such as:
  - Starting material characteristics including purity and traceability
  - Expiration date
  - Lot number
  - Preparation date
  - Methods of measurement and associated uncertainty
  - Actual weights and measurements determined by gravimetric or volumetric measurement
  - Unique identifying number
  - Formula weight
  - Density
  - Half-life of radionuclide(s)
  - Mass and volume of standards
  - Percent of impurities

Receipt, storage, evaluation, use, control, and disposal of all standards as well as documentation of these activities are described in operation-specific SOPs. Additional discussion of standards can be found in section 8.5.4.

4.3 Procurement of Subcontract Laboratory Services

A subcontract laboratory is defined, for the purposes of this QAMP, as a laboratory external to the Quanterra® laboratory network. However, for certain federal programs, a branch location of the Quanterra® laboratory network may also be defined as a subcontractor and require client and agency approval prior to use on a project. If required, Quanterra® will meet those requirements. A subcontracted laboratory will be used only in the event that any of the Quanterra® laboratories do not have the capability or capacity to perform the requested testing, or as directed by the customer. A subcontracted laboratory shall be used only after approval is obtained from the client and the quality of the laboratory is determined to be acceptable according to Quanterra® Corporate SOP Number CORP-QA-0012, "Selection and Evaluation of Subcontractor Laboratories". Once these conditions are met, the subcontract laboratory will be added to an

approved subcontractor list maintained in a database.

Subcontracted laboratories may be removed from approved status on the basis of a failure to perform adequately, as a result of audit findings, nationally recognized or other performance evaluation sample results, or at the request of the Corporate Director of Quality Assurance. The QA staff are responsible for evaluating, approving, and recommending subcontractors. The Project Manager of the primary laboratory is responsible for identifying and initiating prequalification of the subcontract laboratory and managing the subcontractor throughout project implementation.

4.4 Vendor Partnerships

Quanterra® may enter into partnership agreements with vendors under the auspices of the technology organization. The purpose of these vendor partnerships is to standardize instruments, data handling systems, and other products or services which enable or enhance our ability to meet or exceed the requirements of our clients. Vendor partnership agreements must meet the requirements in Section 4.1.

5.0 Documentation and Record Keeping System

5.1 Quality Documents and Records

Quality documents are those which define the objectives, policies, and procedures that ensure the quality of items and services provided by Quanterra®. A system has been designed to revise, distribute, and control quality documents. Quanterra® quality documents are listed in Table 5.1-1 along with their approval requirements.

Records are documents that provide objective evidence of the performance of an item or process. Records are further discussed in Sections 5.5 through 5.7.

5.2 Document Review and Revision

Quality documents have multiple levels of review and approval appropriate for the document. These reviews are demonstrated by the signature of the reviewer on the document. Quality documents are required to be periodically reviewed and, if necessary, revised. The frequency of this review is dependent upon the type of document and regulatory and client requirements. Table 5.2-1 lists the Quanterra® quality documents along with their required frequency of review and the individuals responsible for performing those reviews. In addition to periodic review and revision, quality documents must be revised when the activity, policy, or procedure they describe changes in a significant manner. All changes shall be subject to the same review and approval process. Amendments included in documents shall be clearly marked.

5.3 Document Control and Distribution

Document control is necessary to ensure that:

- the system produces unequivocal, accurate records which document all laboratory activities
- associates have access to current policies and procedures located in or near the area in which work is performed at all times
- only current, authorized versions of policies and procedures are used
- obsolete documents are archived in a manner that allows easy retrieval
- the history of use for particular versions of documents can be reconstructed.

Quality documents that are placed under a controlled distribution include, but are not limited to this QAMP, Quality Policy Documents, and SOPs. Format and control of SOPs are described in Quanterra® Policy Number QA-001, "Standard Operating Procedures." QAPPs are also placed under a

controlled distribution when that document is entirely generated by Quanterra®. These documents shall clearly indicate the effective date of the document and the revision number.

Quality documents are controlled by initially distributing them to the associates who need to be aware of or follow the contained information or procedures. All subsequent revisions or updates to the document are also distributed to the associate. The controlled copy distribution list is maintained with the name of each associate who received a copy along with their controlled copy number. Subsequent revisions or updates to the documents are also controlled and issued based on the distribution list. Obsolete versions of documents are removed from service when new revisions are issued. Further details of responsibilities and systems used for document control are described in facility-specific SOPs. Records of controlled distribution are maintained by Quanterra® and demonstrate that current policies and procedures have been issued to all appropriate personnel.

Controlled documents are marked "Controlled Copy" and are numbered according to the controlled copy distribution list or file. Copies of documents placed under controlled distribution are sometimes released as uncontrolled with the understanding that no further updates or revisions of that document will be issued to that individual document holder. Quality documents are considered proprietary to Quanterra®, but may be issued to outside parties when approved by QA. These are normally issued as uncontrolled copies. Those copies are marked "Uncontrolled" and are not assigned a control number. Uncontrolled copies can also be issued to lab personnel for review prior to implementation. Uncontrolled copies of documents cannot be used to perform work in the laboratory because no updates or revisions will be provided for those copies, and they are not retrieved when new versions are released.

5.4 *Effective Dates and Document History*

The effective date of any quality document, except the QAMP, is the laboratory designated implementation date, the date when controlled copies are distributed and the document is first put into use at a given Quanterra® facility. The implementation date is indicated on the title page of the document. The QAMP implementation date is 60 days after the revision date. Prior to this date, all required reviews of the document are complete; training is completed for personnel who will be using the document; and resources needed to perform the functions described in the document are in place. The document control systems must include a master list or file that identifies the current revision status of quality documents and

records the effective date for each update and version of the documents so that the history of use for each at the facility can be demonstrated.

5.5 Records Management

Information for each business function of the organization is stored appropriately according to its type of information. The record keeping system is intended to allow historical reconstruction of all laboratory activities that produce the resultant analytical data whether manual or computerized. All records, certificates, and project reports shall be safely stored as electronic or hardcopy, held secure, and in confidence for the client. Details of the records management program are contained in the Quanterra® Record Retention Policy No. LEG-004 and supporting documents.

Quanterra® is committed to providing scientifically sound, legally defensible data of known and documentable quality. Legally defensible data are referred to as data which will stand against reasonable adversarial inquiry in the courts-of-law. Data must be supported by a QAPP, client agreements, contractual documents, or the Quanterra® QAMP. Data must also be documented so that the analytical process can be reconstructed.

Project files must be organized so that the project events can be reconstructed if necessary. Accountability for the completeness and accuracy of information must be specified. Supporting information, including data that demonstrates a facility's ability to perform specific analyses, shall be properly maintained.

Records are divided into two distinct types, Quality and Project Records, which are discussed in the following sections. Each laboratory shall have a system in place that provides for appropriate and adequate implementation of these records management requirements.

5.5.1 Quality Records

Quality records demonstrate overall laboratory operation. Examples of quality records include the following:

- Instrument logbooks
- Equipment monitoring records
- Calibration records
- Instrument calibration data
- Maintenance log books
- QC sample data
- Standard preparation logbooks
- Standard certificates
- Standard operating procedures
- Internal and external PE sample results
- Laboratory licenses and accreditations

- Quality reports to management
- Internal and external audit reports
- Nonconformance memos
- Training records.

These records may apply to one or more projects, but in general they are applicable to many projects. Quality records must be properly maintained in the facility files.

5.5.2 Project Records

Project records are documents which are specific to a project or a group of samples within an on-going project. Examples of project records are as follows:

- Chain-of-custody forms
- Raw analytical data
- Final data reports with case narrative and cover letter
- QC and calibration results
- Project-specific nonconformance memos
- Project correspondence including phone logs
- Quotes and Contracts where applicable
- Project-specific QAPPs and SOPs.

Project records shall be properly maintained.

5.5.3 Electronic Data

Where computers or automated equipment are used for capturing, processing, recording, reporting, storage or retrieval of analytical data, the Quanterra's® record handling system is designed to ensure that:

- The requirements in the EPA Good Automated Laboratory Practices (GALP), as expressed in our SOPs are followed
- Computer software is documented and adequate for use
- Procedures are established and implemented for protecting the integrity of electronic media data in terms of data entry or capture, data storage, data transmission, and data processing
- Computers and automated equipment are maintained to ensure proper functioning and the environmental and operating conditions necessary to maintain the integrity of calibration and analytical data
- Security of data, including the prevention of unauthorized access and modification of computer data, is maintained.

Further discussion of control of computer hardware and software is given in the following chapter.

5.6 Storage, Retention and Disposal of Records

When records, as contained in files, are transferred to a records storage area or off-site storage area, they shall be placed in suitable containers and an inventory sheet (hard copy or electronic) prepared by the person submitting the records. The contents of each container will be compared to the inventory sheet and labeled. If there are any discrepancies, the container and inventory sheet shall be returned to the supervisor or Group/Team Leader submitting the records for resolution. In accordance with NELAC requirements, Quanterra® will store all quality records and quality documents, as defined in this Chapter, for a period of five years. Other records and documents will be maintained and disposed of according to Quanterra® Policy Number LEG-004, "Record Retention." All information necessary for the historical reconstruction of data must be retained by the lab. Records which are only stored on electronic media must be supported by the hardware necessary for their retrieval.

5.7 Data Confidentiality

Quanterra® considers the data and associated information for a project to be confidential and the property of the client. In order to preserve client confidentiality, reports and supporting records are only released to third party persons or organizations after consultation with the client and laboratory management. If however directed by courts-of-law or other competent authorities, such as regulatory agencies, Quanterra® will provide required records and notify its clients and provide information as to the identification of the requester and the records that will be released.

The audit reports supplied by federal, state, and local regulatory agencies are public information and can be released without written consent of those agencies. However, specific project audits are confidential and must be approved by the client before releasing them to a third party.

Regardless of confidentiality clauses contained in contractual documents, no information or records are released without first obtaining written approval from the client.

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6.0 Computer Hardware And Software

The purpose of defining controls for computer hardware and software is to protect the integrity of computer-resident data. SOPs shall be put in place to ensure that computer-resident data and programs are accurate, controlled, and secure. The required SOPs and their scope will be presented in the Quanterra® Software Quality Assurance Plan.

Quanterra®'s commitment to meeting good automated laboratory practices is presented in the Quanterra® Corporate Policy Number CORP-IT-013, Software Quality Assurance.

6.1 Use of Hardware

Computer hardware used in the generation, measurement, or assessment of client data shall be of appropriate design and of adequate capacity to function according to specifications. Computer hardware shall be suitably located for operation, inspection, cleaning, and maintenance. The computer shall be installed in accordance with the manufacturer's recommendations. Any changes to the equipment shall be approved by the laboratory Information Technology (IT) representative.

6.2 Security

Procedures shall be in place to insure the integrity of client data. These may include both physical and logical protection and will ensure that access is limited to authorized persons.

Data files will have backup copies made at regular intervals to protect data against accidental loss through a hardware or software failure.

6.3 Use of Software

If computer software is used to acquire, process, or report client data, that software will be tested to ensure that it correctly performs its intended function. Software may be validated or verified, depending upon its complexity, size, and whether it was purchased or developed by Quanterra®. The following definitions are used by Quanterra®:

- Validation - the process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.

- Verification - the process of checking the accuracy of automatically (electronically) calculated information.

6.3.1 Industry Standard Software

Industry standard software programs are defined as those which are purchased and widely used without modification to the program itself. The program is initially verified for use by using test problems with known solutions to demonstrate that the program is operational for the desired application.

All purchased software must be used in accordance with the terms of its software license. Any use of software contrary to its license terms is expressly prohibited by Quanterra®.

6.3.2 Quanterra®-Developed Software

For programs used to process client data and developed within Quanterra®, and externally prepared programs which are modified by Quanterra®, validation or verification must be performed. The process used is dependent upon the function of the software as follows:

- Large complex systems consisting of several programs operating in unison to produce an intended result must be validated.

- For smaller software which only performs numerical manipulation, sample sets of numbers for which results are known should be processed and the results verified. In this case, known results are usually generated by performing hand calculations using the same equations and procedures as the software to verify that the software produces identical results.

- Software which performs as part of instrument operation should be verified as previously described and by processing reference materials through the instrument system. Processed instrument response should be evaluated against expected instrument response and performance.

6.3.3 Control of Software Changes

Changes to software used for processing client data shall be controlled and documented according to the procedures presented in the Quanterra® Corporate Policy Number CORP-IT-013, Software Quality Assurance.

6.3.4 Software Revalidation

Whenever a program is changed, the change will be evaluated to determine if revalidation is necessary. If the software has had features added, previous test problems should be rerun to demonstrate that their function has not been affected. New test problems should be processed, as previously discussed, to verify added performance. If software revision changes the basic operation of the program, complete revalidation of the program may be required.

Spreadsheets and unprotected software used to acquire, process, or report client data must be documented and reverified when changes are made. The test problems used to provide initi

verification shall be reprocessed and the results compared to demonstrate that performance of the software is unchanged.

Laboratory operations is responsible for the generation of the validation and verification data for instrument level software. QA will maintain the necessary documentation. Corporate Information Technology is responsible for generation and maintenance of documentation relating to verification and validation of the Quanterra® LIMS system. This is described in the Quanterra® Corporate Policy Number CORP-IT-013, Software Quality Assurance.

6.4 Documentation

Documentation shall be established for system development, change control, validation, verification, and security. Documentation will be retained according to the Quanterra® Records Retention Policy No. LEG-004.

6.5 Computer Viruses

Quanterra® shall employ the use of anti-virus software to detect and remove viruses from software.

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7.0 Planning

The generation of environmental analytical data is an intricate process. Success is dependent upon the timely execution of interrelated steps, many of which may be project-specific. Quanterra® has an organizational system in place to ensure that all projects are properly planned prior to project initiation, and are monitored for conformance to project requirements during the course of the project. This system ensures that Quanterra®'s clients receive quality deliverables, as well as quality service.

Quanterra® communicates with its clients to identify the client's needs. Project Managers (PMs) work together with Customer Service Managers (CSMs), or designee, to assess and coordinate Quanterra®'s resources. Each client is assigned a single point of contact, usually a PM, to ensure that there is a strong line of communication between the client and Quanterra®. Projects receive technical and QA support at the laboratory or corporate level as needed to ensure that project DQOs are achievable.

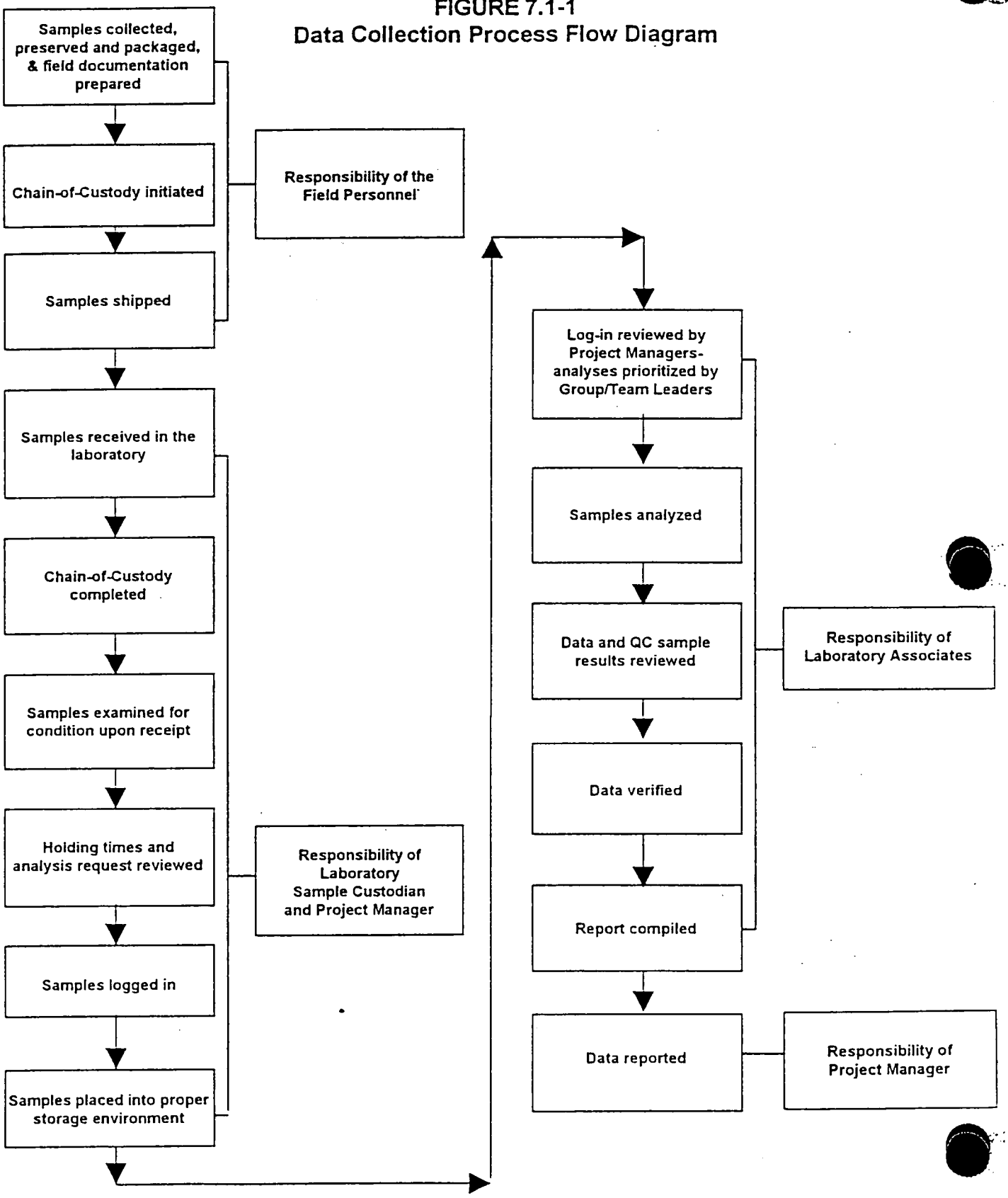
7.1 Data Collection Process

The sample collection and data generation processes are designed to produce analytical data that accurately reflect the nature of the site or sampling point. Figure 7.1-1 shows the sample collection and data collection processes. To ensure our services meet client and project requirements, communication and planning with the client are emphasized. The organization described in Section 7.2 is in place to ensure that these goals are achieved.

7.2 Organizational Responsibilities

Project planning is normally performed within Quanterra® by the operational units. Each operating unit also has the responsibility for customer service within their operating unit. CSMs or designee, Account Managers (AMs), PMs, Operations Managers, and Laboratory Managers play an integral part that will collectively ensure that all projects are thoroughly planned and communicated to all appropriate personnel. Successful project planning and communication of project requirements ensures that the laboratory has facilities and resources to perform the tests required, samples will be handled appropriately and that analytical data will be reported in compliance with project requirements. As a matter of policy, CSMs or designee, AMs, PMs, Lab Managers, and Operations Managers work together to ensure that the following will occur prior to receipt of samples at the laboratory:

FIGURE 7.1-1
Data Collection Process Flow Diagram



- Samples are scheduled for arrival at the laboratory
- All unique project requirements have been identified and communicated to all appropriate personnel
- Standardized client, state, federal, or Quanterra® programs are appropriately selected
- Fully-qualified subcontract laboratories have been selected if needed
- A review has been performed on all pre-project documents such as proposals, contracts, and/or QAPPs to identify range of tests required and within scope of laboratory being used
- All appropriate and required preparations have been made at the laboratory to accommodate or meet project requirements as described in proposals, contracts, and/or QAPPs
- It has been determined that the laboratory has the capability and the capacity to analyze the samples including equipment, staff, space and workload.
- The laboratory has been determined to be able to meet the required sample holding times and is able to report the resulting data within the time line specified by the client
- All known safety hazards associated with the samples have been communicated to all appropriate personnel.

Approval and issuance of a quote, bid or contract document is documentation that this process has occurred. Large projects may require formal management review to assure the above are met.

7.3 Determination of Project QC Requirements

A system must be in place to review project documents [e.g., Request for Proposals (RFP), Request for Quote (RFQ), QAPPs, technical Statements of Work (SOW), or other contractual materials] before a project begins. For larger projects this can involve an inter-laboratory Customer Service Team (CST) (see Section 7.5). Within the laboratory it generally involves the CSM, or PM, or designee working in conjunction with staff from laboratory operations, QA, IT, and management. The goal of the evaluation process is to ensure that client needs and expectations are properly understood, and that the laboratory can meet those requirements before project initiation. Internal and external communications should be in writing to avoid misunderstandings. The CSM, or PM, or designee, with support of others in the laboratory, will work with clients to ensure that project requirements are properly aligned with laboratory capabilities.

When QC requirements are not specified by a client, Quanterra® will follow the requirements given in Corporate Policy Number QA-003, "Quanterra® Quality Control Program" as applicable to the type of project being performed.

7.4 Communication of Project-Specific Requirements

Each operating unit shall use a Quality Assurance Summary (QAS) (example shown in Figure 7.4-1) or an equivalent summary form to document all project-specific requirements. This document is prepared by the PM for all projects prior to sample receipt even if an approved project QA Plan is on file at the laboratory.

Each laboratory shall put into place a hard copy or electronic system that will ensure that the project-specific requirements are disseminated to all appropriate laboratory personnel prior to project initiation.

To further ensure that project-specific requirements regarding analytical methods, QC, and data reporting are thoroughly understood by the laboratory staff, the PMs may conduct project initiation meetings. For large projects that continue over time, the PMs may hold project refresher meetings to ensure that project requirements are continually being met. The PM takes the lead role in implementation of project requirements at the laboratory including changes in project requirements during the course of a project.

7.5 Customer Service Teams

Each operating unit shall establish and maintain long term and substantive relationships with our clients, identify customer needs, and seek to attain value for our customers according to our customers' definitions. An operating unit may create, at any time, client-specific Customer Service Teams (CST). A CST may be created before any discrete projects exist, during on-going projects, or even after the completion of a project. In addition to the PM or customer service representative the team may also contain associates from the operations, technical, QA, IT, contracts, and/or accounting organizations.

7.6 Contingency Planning

An effective QA Program must emphasize contingency planning, actions to prevent problems from reoccurring, and to ensure timely and effective completion of a measurement effort. The following are considered relative to contingency planning:

- Staffing - A primary objective is to ensure that qualified staff are always available to perform the necessary analytical work, regardless of employee turnover, vacation (personal time off), illness, or other absence. Sources of qualified staffing may include temporary agencies specializing in providing technically trained personnel and other Quanterra® facilities. Employee qualification requirements are described in Section 3.0.

FIGURE 7.4-1 (Page 1 of 2)
Example Quanterra® Quality Assurance Summary

Client: _____ Project Code: _____ Contract Name: _____ Site: _____ Project Mgr: _____ **Analysis Type:** ____ Chemical ____ Radiochemical	**RAD SCREEN by:** _____ Code _____ Client-Specific _____ Screens not needed	**SAMPLE DISPOSAL:** _____ Disposal by Lab _____ Return to Client _____ Archive **ANY SPECIAL QC DOCUMENTS?** ____ Yes ____ No	**QAS No.:** _____ Date Initiated: _____ Revision No.: _____ Date Revised: _____

REPORTING

Report to Client: _____ Lab PQL _____ MDL/IDL/MDA _____ < Client DLs _____ Other: _____	**QC Samples to be Reported:** _____ BLK _____ MS _____ LCS _____ MSD _____ DUP _____ Other: _____	**Report Type:** _____ Certificate of Analysis _____ CLP _____ Other: _____	**Report Grouping:** _____ SDG _____ Chain-of-Custody _____ Analytical Batch _____ Other: _____

Report Deliverables: ____ No. of Hard Copies to: _____ ____ Electronic Data Deliverable to: (Diskette Type: _____) Client: _____ Address: _____ _____ ATTN: _____	**Report Deliverables:** ____ No. of Hard Copies to: ____ Electronic Data Deliverable to: (Diskette Type: _____) Client: _____ Address: _____ _____ ATTN: _____

INVOICING

Invoice to: Client: _____ Address: _____ _____ _____ _____ ATTN: _____	Supporting Documentation: _____ Certificate of Analysis _____ RFA/COC _____ Screening Records _____ Other (list): _____ _____	No. of Invoices: _____ Special Instructions: _____ _____ _____ _____

ADDITIONAL COMMENTS OR INSTRUCTIONS:

_____

_____

Project Manager's Approval: _____ Date: _____

FIGURE 7.4-1 (Page 2 of 2)
Example Quanterra® Quality Assurance Summary

Client: _____
Project Code: _____
QAS No.: _____ Revision No.: _____

Number of Samples Expected by Matrix:
_____ Air (A) _____ Water (W)
_____ Soil (S) _____ Other (O): _____

Analysis/Product Code	* Matrix (circle)	Method Prep/Analysis	QC Samples		Required Reporting Limit/Units/ Report as*	Holding Time (Days)	TAT** (Days)	Radiochemical-Specific	
			Type (circle)	Frequency				Count Time	Sample Size
	A W S O		B LCS DUP MS MSD	per					
	A W S O		B LCS DUP MS MSD	per					
	A W S O		B LCS DUP MS MSD	per					
	A W S O		B LCS DUP MS MSD	per					
	A W S O		B LCS DUP MS MSD	per					
	A W S O		B LCS DUP MS MSD	per					
	A W S O		B LCS DUP MS MSD	per					

* A = "As Is" or D = Dry Weight Basis

**TAT = Turn Around Time

SAFETY HAZARDS: Chemical? _____ No _____ Yes: Define: _____
Radioactive? ____ No ____ Yes:
Isotopes Expected: _____ RSO Approval (sign/date): _____

Special Instructions: _____

Comments: _____

- Redundant Instrumentation or Equivalent Methods - At most locations, duplicate instrumentation may be available to ensure uninterrupted work flow. A laboratory may also choose to lease equipment when there is a sufficient time window prior to arrival of samples. However, in circumstances where a catastrophic instrument failure occurs, alternative but equivalent methods may be recommended to the client for approval and implementation.

- Preventive Maintenance - Quanterra®'s preventive maintenance program is designed to minimize analytical instrument malfunctions, permit simple adjustments, and to ensure fewer and shorter breakdowns of critical analytical equipment. (See Section 8.11, "Preventive Maintenance and Service".)

- Network Laboratories & Subcontractor Laboratories - To support the laboratory during peak periods or in the event of a critical instrument malfunction, Quanterra® has the capability to arrange the use of other network laboratories or qualified analytical laboratories as subcontractors for short-term backup analytical support. Through an extensive process, QA personnel evaluate, identify, and select qualified analytical laboratories before an analytical contract is awarded. In order to qualify, a subcontractor laboratory must pass this evaluation. Furthermore, any use of a subcontractor laboratory is approved by the client prior to award of a contract or sample shipment for existing contracts.

- Uninterruptable Power Supply - An Uninterruptable Power Supply (UPS) system which provides line conditioning and backup power to the LIMS computer system/server. This contingency plan allows sufficient time for the main computer system to be shut down and for data archival. All electronically generated data that are stored on the main computer system and on the individual personal computer (PC) hard drives are backed up at regular intervals. In the event that the main laboratory computer system fails, the analytical data can be retrieved from the PC hard drives.

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8.0 Work Processes and Operations

Much of the environmental project activity is planned and designed externally to the laboratory or field operation and presented in the form of a contract, work plan, or QAPP. Laboratory and field activities are in turn planned, implemented, and assessed to meet client requirements according to approved procedures and methodologies. The QAMP provides the systems to document and implement these activities. The execution and assessment of the implemented operational systems are detailed in Quanterra® Corporate or Operation-specific SOPs. The entire process is assessed on a regular basis for conformance to prescribed requirements.

Standard practices for Quanterra® operations are detailed in this section. Specific project or program requirements which differ from those described here can be met. These must exist in approved contracts, work plans, or QAPPs.

In the Facility Specific Appendices are lists of the major equipment and floor plan of the facilities available along with the SOP list for the tests the laboratory performs. Calibration/reference measurement standards are detailed in the SOPs.

8.1 Standard Operating Procedures

SOPs are required in all Quanterra® operating units for analytical and administrative activities from the receipt of samples in the laboratory through analysis, reporting, and subsequent sample disposition. Training, health and safety procedures, QC, method procedures, and instrument and equipment calibrations are included in SOPs. Preprinted forms, either standardized across Quanterra® through a Corporate SOP requirement, or shown as an example form when not standardized, are included in SOPs as appropriate. Standard SOP formats for all activities related to the generation and reporting of data are discussed in the Quanterra® Quality Policy Document No. QA-001, "Standard Operating Procedures". SOPs shall be reviewed by technically-qualified associates. SOPs are controlled documents and are distributed and maintained as described in this policy. SOP requirements for approval and frequency of review are given in Tables 5.1-1 and 5.2-1.

8.2 Analytical Methods

Whenever possible, Quanterra® operations use industry- and regulatory agency-recognized analytical methods from source documents published by agencies such as the Environmental Protection Agency (EPA),

Department of Energy (DOE), the American Society for Testing and Materials (ASTM), and the National Institute for Occupational Safety and Health (NIOSH) as described in Quanterra®'s SOPs. The analytical methods performed by Quanterra® are given in Section 4 of the Facility Appendix for each laboratory. A list of methods routinely performed by Quanterra® is given in Table 8.2-1.

Method performance data (i.e., detection limits, precision and accuracy data) are developed by the laboratory operations staff. The operations staff and the QA staff will evaluate and must approve the performance data before a methodology is performed routinely. The method must also be described and documented with an SOP. In the event that the laboratory staff is required to modify a procedure to meet a unique project requirement, the client will be notified to obtain written approval prior to implementing the change.

All SOPs contain Quanterra®'s interpretation of the published methods. Significant modifications to the published method are described in the SOP. Operations are performed as described in these SOPs. Changes in procedure which may occur due to sample matrix or other events shall be documented in the project-specific case narratives, nonconformance memos (NCMs), or in QAPPs.

8.3 Data Quality Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994)[1]. Typically, DQOs are identified during project scope and the development of sampling and analysis plans. In this QAMP, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. The EPA has established six primary analytical DQOs for environmental studies. These DQOs are precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. Quanterra® incorporates numerous QA and QC samples to

[1] " Guidance for the Data Quality Objectives Process", EPA 600/R-96/005, September 1994.

obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA/QC samples and their applications, described in Section 8.4, are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield monitors.

8.3.1 Precision And Accuracy

Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. Table 8.6-1 illustrates the formulae used to calculate units of precision (i.e., RSD and RPD).

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

The precision and accuracy DQOs that are to be used in evaluating inorganic, organic, and radionuclide constituents at Quanterra® are provided in Tables 8.4-5, 8.4-6, and 8.4-7, method-specific SOPs, and in the documentation for the analytical method of interest.

Precision and accuracy are determined, in part, by analyzing data from matrix spike and matrix spike duplicates, unspiked duplicates, LCSs, and single blind audit samples. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty. A description of these QC samples is provided in Section 8.4.

8.3.2 Completeness

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed.

8.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At each Quanterra® laboratory, every effort must be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

8.3.4 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

8.3.5 Method Detection Limits

It is Quanterra® policy to strictly follow the specification in the U.S. EPA 40 CFR Part 136 Appendix B in determining MDLs for chemical tests. The Quanterra® requirement for this procedure is further detailed in Quanterra® Policy Number QA-005 entitled "Determination of Method Detection Limits for Chemical Tests." This policy requires that the MDLs be determined for each analyte of interest representing the aqueous and solid matrices within the capability of the primary analytical methods. Following the EPA's guidelines, Quanterra® does not provide MDLs for each instrument representing a group of constituents using the same analytical techniques. This policy is based on the statement in the scope and application of the CFR procedure which states that "The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this the procedure was made device- or instrument-independent." Exceptions to this approach are described in Quanterra® Corporate Policy Number CORP-QA-005, "Determination of Method Detection Limits for Chemical Tests". MDLs determined by each laboratory are listed in Section 5 of the Facility Appendix to this QAMP.

8.3.6 Instrument Detection Limits

Instrument Detection Limits (IDLs) are required to be performed quarterly for metals constituents and cyanide when analyses are

performed in support of Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) activities or when the USEPA CLP SOW protocol is required. The requirement for this procedure is described in the Quanterra® Policy Number QA-014, "Determination of Instrument Detection Limits."

IDL samples are introduced at a later stage of the analytical process so that the IDL is a more direct indication of instrument sensitivity. The primary distinction is that the IDL estimates the detection limit of the instrument under ideal conditions, whereas the MDL estimates the detection limit in more-practical terms by subjecting a known concentration matrix to the total method process. IDLs are not required by the SW-846 methods, except for method 6020 where the IDLs are a requirement.

When required, IDLs will be performed in accordance with the procedures defined in the applicable USEPA SOW, ILMO3.0 or subsequent versions, and Quanterra® Policy Number QA-014, "Determination of Instrument Detection Limits".

Prior to acceptance and use for reporting purposes, all data from detection limit studies and reporting limits must undergo technical review and approval by the laboratory management and QA staff.

8.3.7 Reporting Limits

Two reporting limit conventions are used within Quanterra®: the Reporting Limit (RL) and the Project-Specific Reporting Limit (PSRL). Quanterra® Reporting Limit (RL) is the lowest level at which measurements become quantitatively meaningful. An RL is greater than the statistically determined MDLs. In some limited situations, higher RLs may be established based on maximum contaminant level (MCLs), applicable or relevant and appropriate requirements (ARARs), or project-specific data quality objectives (DQOs). The Quanterra® RLs and PSRLs are maintained in the LIMS.

Reporting limits are established and modified within Quanterra® according to the Corporate Policy Number QA-009, "Establishment Reporting Limits."

PSRLs are used when project data quality objectives (DQO) require a reporting limit other than the RL. PSRLs tailor Quanterra®'s product to meet customer requirements.

For radiochemistry, whether the net result is negative, zero, or positive, the actual calculated result is reported with its associated

propagated uncertainty. The minimum detectable concentration (MDC) is affected by many factors, such as the length of count, chemical yield, half-life, background of the instrument, counting efficiency, and the matrix interference. The MDC for radiochemical analyses is defined as the smallest amount (activity or mass) of an analyte in a sample that will be detected with a probability beta of non-detection (Type II error) while accepting a probability alpha of erroneously deciding that a positive (non-zero) quantity of analytes is present in an appropriate blank sample (Type I error). MDCs are determined according to Quanterra® Policy Number QA-007, "Determination of Minimum Detectable Concentration for Radiochemical Tests."

8.4 Quality Control Samples

Two types of Quality Control (QC) samples are field QC samples and laboratory QC samples. Field QC samples are collected during the sampling event and are useful in determining sampling precision and accuracy and monitoring for contamination that may occur during collection, transport or storage of environmental samples. Laboratory QC samples are routinely added at the laboratory to the normal sample stream. Successful analysis of these samples demonstrates that the laboratory is operating within prescribed requirements for accuracy and precision. In addition, utilizing matrix-specific laboratory QC samples, information regarding the effect of the matrix or field conditions on the analytical results can be obtained. The following sections describe common field and laboratory QC samples.

8.4.1 Field QC Samples

When field QC sample collection and analysis are required for a project, it is the responsibility of the project sampling supervisor to ensure that this sampling is performed correctly and at the project-required frequencies. Field QC samples may or may not be identified as such to the laboratory and are considered by the laboratory as field samples for the purpose of QC batching, sample preparation and analysis. Field QC sample results are reported in the same manner as actual field samples, unless a specific deliverable is requested by the client. No correction of the analytical data is done in the laboratory based on the analysis of field QC samples.

Field QC sample types, applicability to organic and inorganic analyses, precision and accuracy applications and by whom they are introduced are summarized in Table 8.4-1. The following sections provide descriptions of field QC samples.

8.4.1.1 Trip Blank

Volatile organic samples are susceptible to contamination by diffusion of organic contaminants through the septum of the sample vial. Trip blanks, also referred to as travel blanks, are analyzed to monitor for possible sample contamination during shipment for volatile organics only. Trip blanks are prepared by filling preserved VOA vials (with no headspace) with organic-free water. Trip blanks accompany the sample bottles during collection and shipment to the laboratory and are stored with the samples.

8.4.1.2 Rinsate Blank

A rinsate blank or equipment blank is a volume of rinse solution (deionized, distilled water or organic solvent) used to rinse a sampling tool. The rinse solution is collected after the sampling equipment has been cleaned in order to demonstrate that there is no residual contamination remaining on the tool that would carry over into the next sample.

8.4.1.3 Field Blank

A field blank is a contaminant-free volume of water or soil that is provided by the sample collector to demonstrate the absence of contamination introduced during sampling. Deionized, distilled water or previously-prepared solid material (e.g., Ottawa sand) is placed into sample containers by the sample collection crew, packaged, and shipped with the other field samples.

8.4.1.4 Field Duplicate

A field duplicate sample is a replicate taken from the same sampling event for that location. The field duplicate sample is submitted to the laboratory as a separate sample by the sample collection personnel. Results of field duplicate samples can provide a measure of sampling precision.

8.4.1.5 Field Matrix Spike

A field matrix spike sample is created by spiking target analytes into a sample in the field at the point of sample acquisition. These sample results provide information on the target analyte stability after collection and during transport and storage.

8.4.1.6 Collocated Samples

Collocated samples are independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. Examples of collocated samples include: samples from two air quality analyzers, sampling from a common sample manifold, or

two water samples collected at the same time and from the same point in a lake.

Collocated samples processed and analyzed by the same organization provide intralaboratory precision information for the entire measurement system including sample acquisition, handling, shipping, storage, preparation, and analysis. Both samples can be carried through the steps in the measurement process together to provide an estimate of short-term precision for the entire measurement system. Likewise, the two samples, if separated and processed at different times or by different people, and/or analyzed using different instruments, provide an estimate of long-term precision of the entire measurement system. Collocated samples processed and analyzed by different organizations provide interlaboratory precision information for the entire measurement system.

8.4.1.7 Split Sample

A split sample is a sample divided into two portions at the sampling site. One portion is sent to a different organization or laboratory and subjected to the same environmental conditions and steps in the measurement process as the other portion sent to another laboratory.

A split sample can be divided into portions at different points in the sampling and analysis process to obtain precision information on the various components of the measurement system. For example, a field split sample provides precision information about all steps after sample acquisition including the effects of storage, shipment, analysis, and data processing. Field split sample results may also reflect the degree of sample homogeneity.

8.4.2 Laboratory QC Samples

Laboratory performance QC is required to ensure the laboratory systems (instrumentation, sample preparation, analysis, data reduction, etc.) are operating within acceptable QC guidelines during data generation as required to meet the client's objectives. Laboratory QC samples consist of method blanks (MB), instrument blanks, laboratory control samples (LCS) and calibration verification samples. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. Typically, this includes matrix spikes (MS), matrix spike duplicates (MSD), sample duplicates, and the use of surrogate compounds.

Laboratory and matrix-spike QC sample types are summarized in Tables 8.4-2, 8.4-3

and 8.4-4. In addition, Tables 8.4-5, 8.4-6 and 8.4-7 list laboratory QC samples, acceptance criteria and corrective actions by reference method for inorganic methods, organic methods, and the USEPA CLP Statements of Work respectively. The following sections provide descriptions of laboratory QC samples and their frequency of use. Quanterra® Policy Number QA-003, "Quanterra® Quality Control Program", describes in detail the QC data evaluation process.

8.4.2.1 Quality Control (QC) Batch

The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period. This definition of a QC batch is utilized by Quanterra® unless there is clear regulatory guidance, contract specifications, or differing client requirements that are explicitly documented. Further details and requirements for the application of the definition of QC batch are described in QA Policy Number QA-003.

8.4.2.2 Method Blank

The method blank (MB) is a QC sample that consists of all reagents specific to the method and is carried through every aspect of the procedure, including preparation, cleanup, and analysis. The method blank is used to identify any interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Potential sources of contamination include solvent, reagents, glassware, other sample processing hardware, or the laboratory environment. In general, the method blank is a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples, that is processed as a sample. In the event that no appropriate solid matrix exists, deionized water may be used. The volume or weight of the method blank must be approximately equal to the sample volume or sample weight processed. A method blank shall be prepared with each group of samples processed.

8.4.2.3 Instrument/Calibration Blank

The instrument blank is an unprocessed aliquot of reagent used to monitor the contamination of the analytical system at the instrument. System contamination may lead to the reporting of elevated analyte concentrations or false positive data. The instrument blank does not undergo the entire analytical process and generally consists of an aliquot of the same reagent(s) used for a sample dilution. Instrument blanks are also

referred to as continuing calibration blanks (CCBs).

8.4.2.4 Laboratory Control Sample

A laboratory control sample (LCS) is a laboratory-prepared suitable clean matrix sample that is fortified with target analytes or a solid reference material purchased from an approved vendor. The LCS contains all target analytes specified in the method, and must contain the same analytes as the matrix spike and matrix spike duplicate. For certain regulatory or client programs, an LCS may contain a full list of analytes. However, in these cases, a subset of analytes, as defined by the program, is used to determine the acceptability of a batch of sample data. The LCS recovery data are used to monitor the analytical method performance in terms of analytical accuracy. On-going evaluation of the LCS recoveries demonstrates that the laboratory is performing the method within statistical control (i.e., accuracy and precision) in the absence of matrix interference. The LCS results, coupled with MS data, help determine whether the laboratory performed the method correctly or the sample matrix affected the analytical results. When a laboratory control sample duplicate (LCSD) is required, a percent recovery for each target analyte is calculated, as well as a relative percent difference (RPD) between the LCS and the LCSD.

8.4.2.5 Matrix Spike

A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. MS samples are analyzed to evaluate the effect of the sample matrix on the analytical methodology. MS samples are generated by taking a separate aliquot of an actual client sample and spiking it with the selected target analyte(s) prior to sample extraction. The MS sample then undergoes the same extraction and analytical procedures as the unfortified client sample. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked and not on all samples in the QC batch.

8.4.2.6 Matrix Spike Duplicate

A matrix spike duplicate (MSD) is a second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with the associated sample and MS sample. The results of the MS and MSD are used together to determine the effect of a matrix on the accuracy and precision of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results may have immediate

bearing only on the specific sample spiked and not all samples in the QC batch. When a full-analyte spike is required, a selected list of analytes is used to measure statistical control or matrix effects.

8.4.2.7 Sample Duplicate

A sample duplicate is a second aliquot of an environmental sample taken from the same sample container that is processed identically with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The results are compared to determine the sample homogeneity and the precision of the analytical process.

8.4.2.8 Surrogates

Surrogates are organic compounds that are similar in chemical composition and behavior to the target analytes but that are not normally found in environmental samples. Surrogates are added to all appropriate samples and QC samples being tested for organic analytes to monitor the effect of the sample matrix and the procedure on the accuracy of the process.

8.4.2.9 Analytical Spike

An analytical spike is created by spiking target analytes into a prepared portion (i.e., post digestion) of a sample just prior to analysis. It provides information on matrix effects encountered during analysis such as suppression or enhancement of instrument signal levels. It is most often used in elemental analysis involving various forms of atomic emission or atomic absorption spectroscopy. A single analytical spike serves as a single point application of the "method of standard additions" or MSA.

8.4.2.10 Interference Check Sample

An interference check sample (ICS) is a solution containing known concentrations of both interfering and analyte elements. Analysis of this sample can be used to verify background and interelement correction factors.

8.4.2.11 Internal Standards

An internal standard (IS) is a compound or element with similar chemical characteristics and behavior in the analysis process to the target analytes, but is not normally found in environmental samples. The internal standard is usually added after sample preparation. The primary function of the internal standard is quantitation, however, it also provides a short-term indication of instrument performance. For isotope dilution methods, internal standards are added during sample preparation and are used for quantitation.

8.4.2.12 Radiological QC Samples

The primary QC sample type used for radiological testing to monitor recovery is the yield monitor. The two types of yield monitors are tracers and carriers. A tracer is a radioisotope, usually of the same element and having the same mode of decay as the analyte. A carrier is a non-radioactive solution added to assist in isolating the specific isotope of an element. When standardized, the carrier can also provide recovery information gravimetrically.

Radiological QC samples and their required frequencies are listed in Section 7 of the Facility-Specific Appendix when applicable.

8.5 Data Collection Operations

Laboratory analyses are designed to produce data that are representative of existing conditions present at the time the sample was obtained. The data collection design includes field sampling events, sample handling and custody, analytical operations, data recording procedures, data assessments, data verification, and data reporting requirements and techniques to assess limitations of data use. These operations are discussed in Sections 8.5 through 8.10.

8.5.1 Field Collection and Shipment

In order to provide a sample that most accurately represents the test matrix, field sample collection personnel must abide by the sample collection guidelines and procedures established by involved regulatory agencies. A significant part of the efforts of regulatory agencies include the use of "approved" sample containers, chemical and physical preservation techniques, and observance of specified holding times. It is imperative that all samples be collected and preserved according to the appropriate analytical method specified in the QAPP (if one exists). Although the sampling may be performed by non-Quanterra® personnel, the importance of sampling and transportation of the sample to the laboratory is understood and must be considered during data validation.

Sampling requirements must be communicated to the sampling team prior to field collection.

Field personnel are responsible for labeling each individual sample collected with the following information:

- Project name
- Unique client sample number
- Sample location (including as appropriate: borehole and depth or grid coordinates)
- Sampling date and time
- Sample preservation

- Analysis required.

An overriding consideration for the resulting analytical data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody, and disposal must be documented to accomplish this. Figure 8.5-1 shows an example Chain-of-Custody (COC) form that is used by the Quanterra® laboratory network to document this evidence. Field personnel are responsible for initiating the COC form.

The prompt shipment of samples to the laboratory is necessary to ensure that required holding times are met. Samples should be shipped by an overnight carrier, be hand-delivered, or transported in a manner that assures prompt delivery to the laboratory.

Some sites require an extensive radioactive screening process before a sample may be shipped. In these cases, it is imperative for the Project Manager to maintain good communications with the client to assure proper staffing of the laboratory in response to a decreased holding time.

Radioactive samples that are shipped to Quanterra® radiochemistry operations must be screened upon receipt and found not to contain radioactivity that exceeds the level stated in the laboratory's operation license. Samples received by a Quanterra® facility containing radioactivity exceeding their license limit will immediately be returned to the project site.

8.5.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times

8.5.2.1 Sample Containers

A sample container is defined as the sealed enclosure, usually made of plastic or borosilicate glass that the sample is collected in and stored in until analysis. All sample containers provided by Quanterra® operations for environmental sampling are new, with the exception of some air sampling canisters, which must be recertified before reuse, and demonstrated to be clean for their appropriate use. All documentation certifying sample container cleanliness must be maintained by the laboratory or the vendor and can be provided to the client upon request. The sample containers to be supplied are listed in Tables 8.5-1 through 8.5-5. Sample containers provided to the client by Quanterra® are transmitted under custody.

Quanterra®

FIGURE 8.5-1
Example Quanterra® Chain-of-Custody Form

Reference Document No. _____
Page 1 of _____

Project Name/No.: _____
Sample Team Members: _____
Profit Center No.: _____
Project Manager: _____
Purchase Order No.: _____
Required Report Date: _____

Sample Shipment Date: _____
Lab Destination: _____
Lab Contact: _____
Project Contact/Phone: _____
Carrier/Waybill No.: _____

Bill To: _____
Report To: _____

ONE CONTAINER PER LINE

Sample Number	Sample Description/Type	Date/Time Collected	Container Type	Sample Volume	Preservative	Requested Testing Program	Condition on Receipt	Disposal Record No.

Special Instructions:

Possible Hazard Identification:
Non-hazard ❑ Flammable ❑ Skin Irritant ❑ Poison B ❑ Unknown ❑

Sample Disposal:
Return to Client ❑ Disposal by Lab ❑ Archive _____ (mos.)

Turnaround Time Required:
Normal ❑ Rush ❑

QC Level:

1. Relinquished by: *(Signature/Affiliation)* Date: _____ Time:	1. Received by: *(Signature/Affiliation)* Date: _____ Time:
2. Relinquished by: *(Signature/Affiliation)* Date: _____ Time:	2. Received by: *(Signature/Affiliation)* Date: _____ Time:
3. Relinquished by: *(Signature/Affiliation)* Date: _____ Time:	3. Received by: *(Signature/Affiliation)* Date: _____ Time:

Comments:

8.5.2.2 Shipping Containers

Shipping containers are defined as the sealed enclosure in which the sample containers are stored during shipment from the sample collection site to the analytical laboratory. Shipping containers must be of sufficient number and size to accommodate the samples in an upright condition. Shipping containers must also meet all requirements for the shipment of environmental and/or radioactive samples.

Packaged samples must be shipped to the analytical laboratory in a safe manner that preserves the integrity of the samples. The most common method of sample shipment employs coolers or ice chests that are sealed with custody tape and shipping tape. These coolers must be durable and resistant to crushing during shipment. All coolers must be well maintained and cleaned to prevent cross-contamination of the samples. It is the ultimate responsibility of the person collecting and packaging the sample for shipment to ensure that the shipping containers are clean and functional.

To help prevent sample breakage during shipment, additional consideration must be given to providing shock absorbency to all samples packaged inside the shipping container. Use of bubble-wrap around each sample container is the best way to provide this protection. Foam packing materials and vermiculite are also successfully used.

8.5.2.3 Sample Preservatives

Most analytes have a finite holding time in a given sample matrix. Sample preservation is the chemical or physical means by which samples are treated during and/or following sample collection to aid in the stability of the analytes of interest in that matrix. Sample holding times are also adversely affected when samples are improperly preserved, or shipped unpreserved. The preservation of samples at the time of sample collection will follow the requirements of the analytical methods used. This preservation includes the addition of reagents to deter chemical and biochemical degradation and the maintenance of refrigeration during transit and ultimate storage in the laboratory. The required preservatives for the analysis to be performed on each matrix are included in Tables 8.5-1 through 8.5-5.

8.5.2.4 Sample Holding Times

Holding time is defined as the maximum allowable time a sample can be stored after sample collection and preservation (or laboratory receipt for CLP) until appropriate processing occurs (preparation or analysis). The holding time may vary according to method or client requirements. Tests designated with

holding times as "analyze immediately or ASAP" are considered parameters that should be tested by field personnel or on-site. Each operation has a system in place to ensure that holding times are monitored by each group within the operating unit. It is the responsibility of each Quanterra® associate processing the sample to assure that holding times are met. Quanterra® laboratories are responsible for meeting all holding times for properly preserved samples received within 48 hours of collection or if less than half the holding time has passed. If these conditions are not met, Quanterra® will attempt to expedite sample analysis as soon as possible.

Sample holding times are listed in Tables 8.5-1 through 8.5-5.

8.5.3 Sample Handling

Each Quanterra® laboratory has a facilitly-specific SOP describing sample receipt and log-in in detail. The following sections describe the general policies followed by all Quanterra® laboratories.

8.5.3.1 Sample Receipt

Samples shall be received and logged in at Quanterra® operations by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of gloves, a lab coat, safety glasses, and in some cases a respirator
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the COC
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA samples) and record on the COC
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the COC, immediately initiate a Condition Upon Receipt Report (CUR) or equivalent format,

and notify the PM who in turn notifies the client

- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A CUR or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client shall be notified by the PM or designee of all CURs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CURs must be resolved prior to sample preparation and analysis. The completed CUR form shall be stored in the project file. An example CUR is shown in Figure 8.5-2. The report narrative will include an explanation of sample receiving related anomalies.

8.5.3.2 Sample Log-in

Sample log-in activities at Quanterra® operating units are fully documented in operation-specific SOPs. The following is a general description of the log-in process:

- Enter the samples in the laboratory sample log-in book, and/or the LIMS which contains the following information at a minimum:
  - Project name or identification number
  - Unique sample numbers (both client and internal laboratory)
  - Type of samples
  - Required tests
  - Date and time of laboratory receipt of samples
  - Field ID supplied by field personnel
- Notify the PM and appropriate Group/Team Leader(s) of sample arrival
- Place the completed COCs, waybills, and any additional documentation in the project file.

8.5.3.3 Sample Storage

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination

FIGURE 8.5-2

Example Quanterra® Condition Upon Receipt Anomaly Report (CUR)
Page 1 of 2

CUR No.: _____
Lot: _____

Date Received: _____
Initiated By: _____
RFA/COC Nos.: _____

Client: _____
Project Name: _____

Condition/Anomaly/Variance (select from legend on back):

Client Sample ID	Analysis Requested	Condition (see legend)	Comments/Actions

Corrective Action:

- [ ] Client informed verbally on: _____ By: _____ In writing on: _____ By: _____
- [ ] Sample(s) processed "as is"
- [ ] Sample(s) ON HOLD until further notice
- [ ] Other action required (see above comments)
- [ ] No action required.

Sample Control Reviewer: _____ Project Manager: _____

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

FIGURE 8.5-2
Example Quanterra® Condition Upon Receipt Anomaly Report (CUR)
Page 2 of 2

Legend:

Cooler:
- 1a Not received, COC available
- 1b Leaking
- 1c Other: _____

Temperature:
- 2a Temp. Blank: _____
- 2b Cooler Temp: _____
  (cooler temp should only be used if there is no Temp. Blank)

Containers:
- 3a Leaking
- 3b Broken
- 3c Extra
- 3d No labels
- 3e Headspace (VOA only)
- 3f Other: _____

Samples:
- 4a Samples received but not on COC
- 4b Samples not received but on COC
- 4c Holding Time Expired
- 4d Sample Preservative: _____
- 4e Other: _____

Custody Seals:
- 5a None
- 5b Not intact
- 5c Other: _____

Chain of Custody (COC):
- 6a Not relinquished by client
- 6b Incomplete information
- 6c Other: _____

Container Labels:
- 7a Doesn't match COC
- 7b Incomplete information
- 7c Marking smeared
- 7d Label torn
- 7e Other: _____

Other (8): _____

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

of samples. Low-level and high-level samples, when known, must be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests should be monitored twice per month by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial)

- Security of samples within the laboratory.

The requirements listed in Tables 8.5-1 through 8.5-5 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. Quanterra® operations will assign individuals the responsibility of notifying the Operations Manager and Group/Team Leaders if there are any samples which must be analyzed immediately because of holding time requirements.

8.5.3.4 Internal Sample Chain-of-Custody and Interlaboratory Transfers

Sample custody within Quanterra® laboratories is described in operation-specific SOPs. The sample custody documentation shall include the following minimum requirements:

- Name of associate taking custody of the sample from the sample storage area for preparation or analysis
- Dates sample removed from and returned to the sample storage area
- Identification of tests to be performed on the sample aliquot(s) selected by the associate
- Sample matrix
- Laboratory sample numbers
- Sample storage location.

Additional custody records can be provided by a facility at the specific request of the client. Access to all Quanterra® facilities is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

Samples transferred to a different laboratory than the original receiving facility are transferred under chain-of-custody (COC). The COC is maintained whether the laboratory is another Quanterra® facility or a subcontracted laboratory. If the entire sample volume is transmitted, the original copy of the client's COC form will be used to document the relinquishing of the sample and will accompany the sample to its destination. A copy of the completed COC form shall be retained in the laboratory project file. In the case where an aliquot of a sample is shipped

from the laboratory, a new COC will be generated by the laboratory and shipped with the sample aliquot. The original COC will be retained in the project file at the site holding the original sample container.

Samples are not transferred to other Quanterra® facilities or to subcontractor laboratories without prior approval of the client.

8.5.3.5 Sample Disposal and Return Chain-of-Custody

After the requested analyses on the samples have been completed, any remaining portions of the samples will be maintained by the sample custodian until the samples are disposed of or returned to the client. The disposal of each sample is recorded on the client's COC form, in LIMS, or referenced in the project file. Sample disposal procedures and documentation are described in operation-specific SOPs. Quanterra®'s routine sample retention period is at least thirty days after the analytical report is issued to the client, unless otherwise specified by the client.

For Nuclear Regulatory Commission (NRC) or state licensed laboratories, a real-time inventory of all radioactive isotopes contained in the laboratory (including radioactive samples), as required by the NRC or state, is maintained by the Radiation Safety Officer (RSO). If the quantities of radioactive materials in-house approach the limits stipulated by the laboratory NRC or state license, appropriate action must be taken to ensure the licensed level is not exceeded. This may involve returning samples to clients immediately.

If samples are returned to the client rather than disposed of by the laboratory, the original COC is used to document custody transfer back to the client from the laboratory. A copy of the completed COC is retained in the laboratory project file.

8.5.4 Calibration Procedures and Criteria

All equipment and instruments used at Quanterra® operations for quantitative measurements are controlled by a formal calibration program. Calibrations may be periodic or operational. These are described in operation-specific and corporate SOPs. At a minimum, these procedures shall include:

- Instrument to be calibrated
- Reference standards used for calibration
- Calibration technique (e.g., linear, quadratic)

- Acceptable performance tolerances and corrective actions required if specifications are not met
- Frequency of calibration
- Calibration documentation requirements.

Whenever possible, recognized procedures such as those published by ASTM or the USEPA or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operation error on the quantities measured.

8.5.4.1 Physical Reference Standards

Physical reference standards associated with periodic calibrations include weights for calibrating balances and certified thermometers for calibrating working thermometers. Whenever possible, physical reference standards shall be calibrated by a body that can provide traceability to nationally or internationally recognized standards. If these standards are not available, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration procedures and shall be stored separately from equipment used for analysis.

8.5.4.2 Chemical Reference Standards and Reagents

Chemical reference standards are generally associated with operational calibration. These standards include reference materials traceable to recognized standards suppliers. This may include vendor-certified materials traceable to national or international standard reference materials (e.g., NIST or A2LA).

All chemical reference standards maintained in the laboratory for use in calibrations (or as QC spiking solutions) shall be labeled or referenced to appropriate documentation with the following information at a minimum:

- A unique identification including concentration (solutions containing several analytes can be identified such that the solution constituents and concentrations can be referenced to a logbook)
- Medium prepared in
- Preparation date
- Expiration date
- Initials of preparer.

Vials containing standard solutions that are not large enough to accommodate labels listing the above information may be referenced to a laboratory logbook or notebook entry. The expiration date of the working standard must not exceed the expiration date of the original material.

8.5.4.3 Standard Verification

When possible, reference standards are purchased from a Quanterra® preapproved vendor. Standards are verified before use by the vendor or Quanterra® where applicable. Prior to use, the laboratories must confirm that the lot they received from the vendor was approved. Special standards that are obtained from another source must be independently verified at the lab. Verification of a reference standard from neat materials is also necessary.

To extend the use of an expired standard, reverification is necessary provided that new analysis produces acceptable data. The verification of an expired standard is performed against a current, independent standard reference material by analyzing within a valid calibration and QC.

Stock and working standards are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard solutions. Standards are always stored separately from samples.

An independent standard is used to verify initial calibrations. An independent standard is defined as a standard composed of the same target constituents as, but from a different source than those used in the standards for the initial calibration. An independent standard may be a laboratory-prepared or a certified independent standard solution(s). Independence of reference material can be achieved by: (1) purchasing reference materials from two separate vendors, (2) using a different lot, or (3) having two separate individuals prepare the calibration and verification standard solutions if independent sources are not available for neat standards.

Records for all purchased standards and reagents shall include the date of receipt, the date opened, and, where applicable, the expiration date.

8.5.4.4 Periodic Calibration

Periodic calibration is performed at prescribed intervals. In general, equipment which can be calibrated periodically is a distinct, singular purpose unit and is

relatively stable in performance. These include balances, micropipettors, counters, thermometers, refrigerators, freezers, and ovens. Equipment employed at Quanterra® operations requiring periodic calibration are listed along with their respective calibration requirements in Table 8.5-6. Each Quanterra® operating unit has SOPs in place for the calibration of this equipment if in use at their location.

8.5.4.5 Operational Calibration

Operational calibration is routinely performed as part of instrument usage, such as the development of a standard calibration curve. The accuracy of initial calibrations are to be verified prior to sample analysis through the use of an independent standard in situations where the source method requires calibration verification. A laboratory control sample (LCS), prepared from an independent standard, may also be used for calibration verification. Detailed requirements for operational calibration are contained in method-specific SOPs. A summary of the various operational calibrations performed at Quanterra® operations is shown in Tables 8.5-7 through 8.5-9.

8.5.4.6 Calibration Failure

Equipment or instruments that fail calibration or become inoperable during use shall be tagged to indicate they are out of calibration. Such instruments or equipment shall be repaired and successfully recalibrated before reuse. Following recalibration or verification, back to control will be documented in the injection/run log and/or maintenance logbook through the routine identification of the required calibration runs specified by the standard operating procedure. The procedure for tagging out such instruments or equipment is described in the Corporate SOP Number CORP-QA-0010, "Nonconformance and Corrective Action."

Recalibration may occur more frequently than scheduled. At any time, if equipment calibration becomes suspect, it shall undergo a calibration check to determine whether the current calibration is still acceptable or if recalibration is required.

8.5.4.7 Calibration Records

Calibration shall be documented for each piece of equipment subject to calibration. All calibration records (periodic and operational) directly affect data and may not be limited to one project. These records shall be stored in either the quality records or the associated project files. Project files that include sample data shall either include the calibration records or include reference to them.

8.6 Quality Assessment

The effectiveness of the QA practices at a laboratory is measured by the quality of data generated by the laboratory. Each Quanterra® operating unit shall establish, implement and document procedures to detect, prevent, and correct quality problems and to ensure quality improvement. Items and processes that do not meet established requirements must be investigated to determine their cause. Improvements must be implemented in the operations which will prevent a recurrence of these quality problems and provide overall quality performance. All phases of laboratory work should be designed with the objective of preventing problems and improving quality on a continuous basis.

8.6.1 Data Quality Assessment

Data quality is judged in terms of precision, accuracy, representativeness, completeness and comparability. The areas of representativeness, comparability, and completeness for an overall project, inclusive of sampling issues, may be beyond the control of the laboratory. The elements over which the laboratory has direct control are precision, accuracy, and completeness relative to analytical testing results.

Precision and accuracy assessments are made as part of the evaluation of laboratory QC data generated during sample preparation and analysis. The QC samples employed at Quanterra® as part of routine sample analysis are summarized in Section 8.4 of this document. Table 8.6-1 shows the precision and accuracy measurements employed by Quanterra®. Analytical method SOPs and Quanterra® Policy Number QA-003, "Quanterra® Quality Control Program", include information on requirements for the type of QC samples, frequencies, and acceptance criteria. Additionally, the SOPs and Policy describe the appropriate actions to be taken when a QC sample result does not meet acceptance criteria.

8.6.2 Statistical Evaluation of Data

In-house limits for all QC data must be evaluated at least annually and compared to the limits published in the methods for applicable matrices. Method limits will be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These

additional requirements shall be documented in a QAPP or QAS.

Precision and accuracy measurements employed by Quanterra® are shown in Table 8.6-1. Calculated results of these QC samples are evaluated using statistical tables or control charts.

8.7 Data Recording Procedures

To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Quanterra® Corporate SOP Number QA-008, "Data Recording Requirements".

8.8 Data Reduction and Verification Procedures

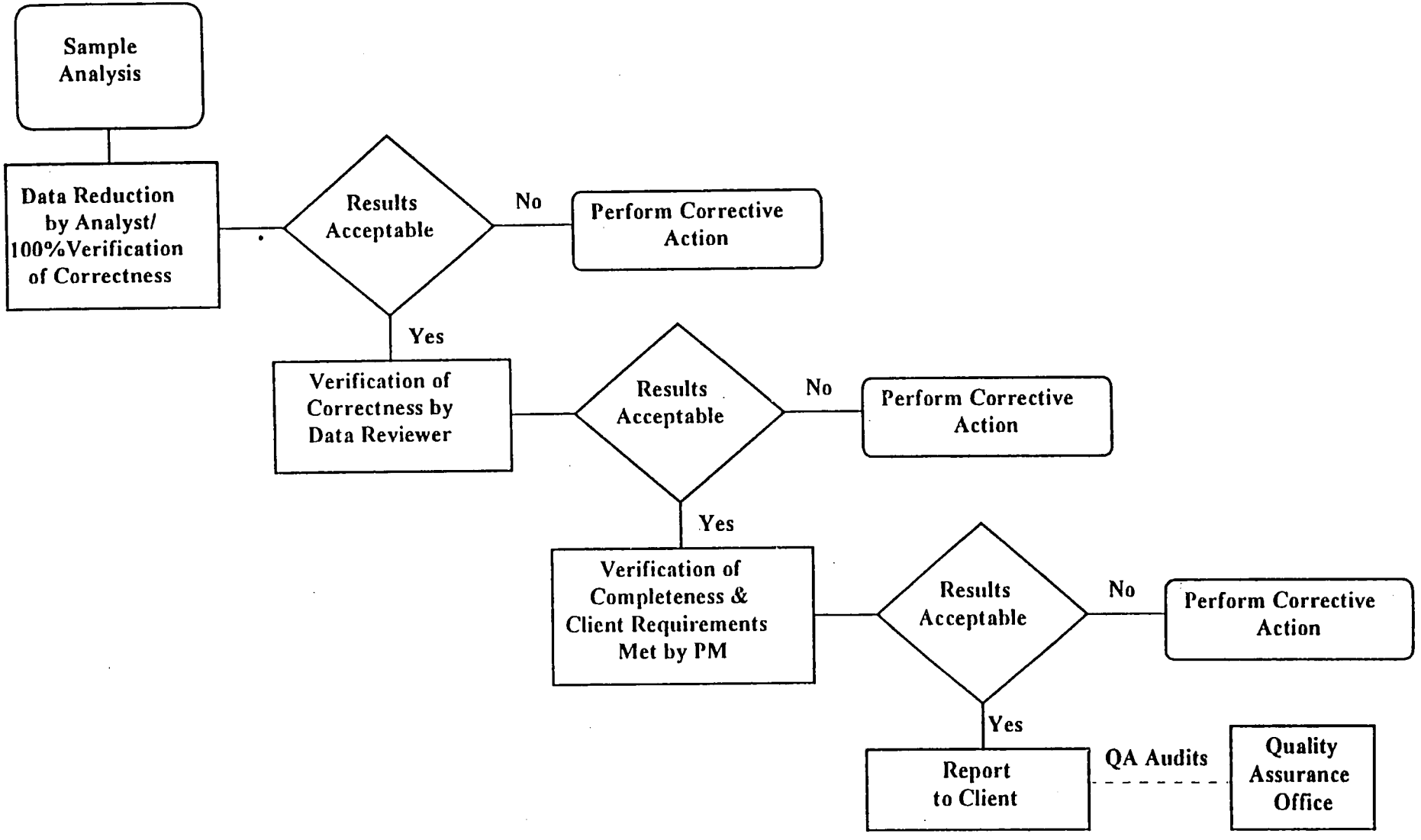
Data review procedures, defined as a set of computerized and manual checks applied at appropriate levels of the measurement process, will be clearly defined for all measurement systems in SOPs. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100 percent review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed. This procedure is outlined in Figure 8.8-1.

8.8.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.

FIGURE 8.8-1
Data Reduction, Verification, and Reporting



- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project records
- Proper documentation procedures have been followed
- All nonconformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to produce good chromatography, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Quanterra Policy Number QA-011, "Acceptable Manual Integration Practices".

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation and processed, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units

- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst. Data review requirements are described in the Quanterra Policy Number QA-012, "Technical Data Review Requirements".

8.8.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, supervisor, or designee. This check is performed to ensure that level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. This review includes an evaluation of all items required in the raw data package. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and specific instructions given on the project QAS or equivalent summary form

- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo

- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct

- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist. Data review requirements are described in the Quanterra Policy Number QA-012, "Technical Data Review Requirements".

8.8.3 Completeness Verification

A third-level review is performed by the PM. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)

- Every parameter or target compound requested is reported with either a value or reporting limit

- The correct units and correct number of significant figures are utilized

- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory

- The final report is legible, contains all the supporting documentation required by the project, and is in either the standard Quanterra® format or in the client-required format.

A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

8.9 Data Reporting

8.9.1 Data Reports

Quanterra® is capable of developing a variety of data deliverable reports. In general, Quanterra® reports contain:

- Cover Letter/Narrative - Information on sample types, tests performed, any problems encountered, and general comments are provided.

- Analytical Data - Data are reported by sample or by test with the appropriate significant figures and reporting limits, and

have been adjusted for dilution, if appropriate. Pertinent information including dates sampled, received, prepared, extracted, and analyzed are provided.

- Laboratory Performance QC Information - The results of LCSs and method blanks analyzed with the project are listed. Any data or QC anomalies are discussed in the narrative.

- Matrix-Specific QC Information - Results of any sample duplicates and MS/MSDs analyzed with the samples as batch QC are reported. Other project-specific QC requested by the client are also reported. The results include supporting information such as amount spiked, percent recovery, or percent difference/RPD.

- Methodology - Reference for analytical methodology used is cited.

- Other Deliverables - Other deliverables available include disk deliverables, electronic data transfer, sample raw data packages, complete deliverable packages, and custom report formats.

8.9.2 Verbal Results

Quanterra®, as a policy, discourages the release of data verbally or without full data review. If however, the client requests analytical results to be communicated verbally or by facsimile prior to final review, they must be clearly identified as "Preliminary" results. The client must understand that the data have not undergone the required levels of review and may potentially change.

8.9.3 Reporting Analytical Results

Sample results are reported according to analytical method SOPs or client specifications. Normally, the laboratory uses the Quanterra® Reporting Limit (RL) at which any analyte of interest detected at or above that level is reported as a positive value and any analyte of interest not detectable or detected below that level is reported as "not detected" at the RL. The laboratory will normally report results within the calibration, however, any reported results outside of the calibration range will be documented in the final report.

In some cases a contract, QAPP, or documented client request may require the laboratory to report sample results in a specified manner. Some examples are given below:

- The laboratory may be requested to report all analytes of interest that are less than the laboratory's RL but are greater than the MDL. This data will be flagged with an appropriate qualifier. (See precautions in "Establishing Reporting Limits", Policy Number QA-009.)

- The laboratory may be requested to report any tentatively identified compounds (TICs). This data will be flagged with an appropriate qualifier.

- The laboratory may be requested to report sample results using an RL that is higher than their normal level. In this case, only the analytes of interest found at or above that level would be reported as positive values.

In this case, the laboratory will state the PSRL rather than the RL. All analytes of interest not detected or detectable below that level would be reported as "not detected" at the PSRL.

In these types of cases, the laboratory must include documentation in the project file that supports the reporting procedure employed.

It is the responsibility of the laboratory to provide for a reporting system that assures that any problems associated with an analysis are properly documented on a nonconformance memo, communicated to the appropriate Quanterra® associates, and addressed appropriately in the data report. If, after issuance of a report, Quanterra® observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified.

8.10 Data Validation

Data validation for Quanterra® refers to data reviews conducted in accordance with the

USEPA CLP "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses", or modifications thereof, for non-CLP type analyses.

This form of data validation provides an impartial evaluation of the laboratory's results. Data validation may be requested by the client for a percentage of data and is usually performed by a third party, one which was not involved with the sample analysis. Qualifiers are assigned to data, when required, according to the requirements of the data validation protocol being used.

8.11 Preventive Maintenance and Service

Facilities, instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within Quanterra®, preventive maintenance, coupled with vendor service agreements, is an organized program of actions taken to maintain facilities and equipment in control.

8.11.1 Analytical Instrumentation and Equipment

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

At each laboratory each instrument or piece of equipment shall be uniquely identified. Each operating unit shall maintain the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit.

The records of routine maintenance and non-routine maintenance shall include at a minimum:

- Name and serial number of the item or equipment
- Details of maintenance performed
- Dates and results of recalibrations/reverifications indicating back to control
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative.

Any item or equipment that does not perform to specifications or defective shall be taken out of service, clearly identified and segregated until it has been repaired and shown by calibration/verification to perform satisfactorily.

Quanterra® documents and describes in detail instrument and equipment preventive maintenance procedures and requirements in operation-specific SOPs.

8.11.2 Facilities

Another important aspect of the laboratory operation is the existence and maintenance of adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme temperatures, waste disposal, and a source of stable power.

The maintenance and use of these facilities and proper operations are described in the Quanterra® Chemical Hygiene Plan (CHP). The Laboratory Manager has responsibility for ensuring a properly maintained facility. The Laboratory Manager also has the responsibility for ensuring that samples are stored properly without contamination, work areas are equipped with adequate bench, hood and operational space, and the areas are free from chemical and radiological contamination that may affect analytical results.

8.11.3 Frequency of Maintenance

The frequency of maintenance must consider manufacturer's recommendations and previous experience. Frequency of preventive maintenance along with the recommended preventive maintenance schedules are given in Tables 8.11-1 through 8.11-27 for analytical instrumentation and equipment or defined in operation specific routine maintenance SOPs. These schedules are the recommended default if

not otherwise specified by the manufacturer, SOP, or Facility-Specific Appendix for the laboratory. Frequency of maintenance for the facility systems is documented in the Quanterra® CHP.

8.12 Other Requirements

8.12.1 Water

High purity water (i.e., ASTM reagent grade or equivalent water) will be used in all metals, radiological, wet chemistry, and organic analyses. Demonstration of contaminant-free water is shown through the analysis of method blanks consisting of the reagent water on a daily basis for the analyte of interest. This water is obtained by the use of either a commercial ion-exchange deionizing, distillation, or reverse osmosis unit plus an appropriate polishing unit. The resulting water has a maximum conductivity of 1.0 umho-cm at 25°C or a minimum resistivity of 1.0 Mohm at 25°C. Conductivity or resistivity will be monitored and documented daily or on each day that water is dispensed for analytical use.

For volatile analyses the water may be further purified by purging with an inert gas before use to remove potential traces of organic solvents.

Water monitoring procedures used by Quanterra® operating units are detailed in operation-specific SOPs.

8.12.2 Compressed Air and Gases

Ultra high-purity compressed gases from preapproved vendors or in-house gas generators will be used when required for instrumentation. These air and gases must meet the requirements and specifications of the analytical methods performed. In-line filters will be used when appropriate to minimize contamination and moisture from the gases.

8.12.3 Glassware Preparation

Glassware preparation procedures implemented at Quanterra® operating units are designed to ensure that contaminants are not introduced during sample analysis. Procedures describing glassware preparation are detailed in operation-specific SOPs.

8.12.4 Chemical Storage

Storage of chemicals shall be conducted in a manner to minimize the potential for fire or release of hazardous material resulting from an unplanned chemical reaction. Refrigerators used for storing flammable liquids must have spark-free interior construction. Flammable solvents shall be stored in appropriate cabinets meeting all necessary codes. All chemicals are stored according to chemical compatibility. Further details regarding chemical storage are provided in the Quanterra® CHP.

8.12.5 Waste Disposal

Laboratory wastes shall be disposed of safely and in a manner consistent with applicable regulations. The Laboratory Manager or designee is responsible for the development, implementation, and maintenance of site-specific procedures that will document all aspects of the disposal program. These procedures and the training requirements are described in the Quanterra® CHP.

8.12.6 Facility Security

Each Quanterra® laboratory is a limited access, secure facility. To ensure that only authorized personnel are able to enter the building from an entrance that is not monitored, entry into each building is limited in one or more of the following ways at a minimum:

- The use of key pads or electronic locks activated by swipe or magnetic cards which are issued only to authorized personnel
- Locking doors and issuing keys only to authorized personnel
- Alarm systems to detect unauthorized entrance
- During business hours, entry is possible only through the main entrance. This entrance is monitored at all times, usually by a receptionist. All guests are required to sign in by using a visitor logbook.

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9.0 Quality Assessment and Response

9.1 Nonconformance and Corrective Action

9.1.1 Nonconformance

A nonconformance is an unplanned deviation from an established protocol or plan. The deviation may be the result of Quanterra's® actions as a systematic error, then termed a deficiency. A single isolated event or event beyond the control of Quanterra® is then termed an anomaly.

Nonconformances can be identified on the basis of internal or external systems or performance audits, sample processing, routine calibration and monitoring of analytical and support equipment, or QC sample analyses. The Operations Manager, Project Manager, QA Manager, Area Leader, and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected, the issue is immediately brought to the attention of Corporate QA and senior management.

9.1.2 Corrective Action

Corrective actions are measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence. Corrective actions should be timely, determine the root cause, and evaluate any propagation of the error or problem. Whenever a systematic error is discovered that affects the accuracy or defensibility of results reported to Quanterra®'s clients, client notification will be part of the corrective action. Corrective actions should be implemented with an understanding of the technology and work activities associated with the quality element, with appropriate training of Quanterra® associates and vendors, and should be monitored for progress and success.

Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented properly. On-the-spot actions are used to correct minor problems, such as recalibration, retuning, or a minor repair (e.g., replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used by an analyst. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Similarly, routine instrument maintenance, malfunctions, and power failures are also documented in the appropriate instrument maintenance logbooks. These events do not require a formal NCM process. Corrective actions specific to analytical methods are discussed in the operational-specific SOPs.

9.1.3 Responsibilities

A detailed description of the responsibilities associated with nonconformance and corrective action, as well as the procedures to be followed, is provided in the Quanterra® Corporate SOP Number CORP-QA-0010, "Nonconformance and Corrective Action."

9.1.4 Nonconformance Memo

As defined in the SOP, deficiencies and anomalies for all activities other than sample log-in or matrix related incidents shall be documented on a nonconformance memo or via an electronic process that meets NCM requirements as approved by QA. Deficiencies and anomalies may be documented on separate systems or forms. A log or computerized data base will be maintained for all nonconformances determined to be deficiencies. Deficiencies will be examined for trends periodically, and this evaluation will be documented and reported to management. A copy of the nonconformance memo will be kept in the project files along with the data it refers to. A copy shall also be kept in the quality files.

9.2 Audits

Audits of Quanterra® laboratories are performed to assess the degree of adherence to established policies, procedures, and standards. These assessments are conducted internally by Quanterra® personnel, who are independent of the area being evaluated, and externally by clients and regulatory agencies. Audits can identify areas for improvement with regard to compliance with policies, procedures, and standards. Audits also provide a means for correction prior to system failure. The following types of audits and assessments are performed at Quanterra® laboratories:

- Performance Audits
- Systems Audits or Internal Systems Evaluations
- Data Audits
- Spot Assessments.

Internal systems audits or evaluations are generally conducted by QA staff, although periodic self-audits may be conducted by the operational units. Audits and assessments are generally conducted through the use of checklists and appropriate reference documents. Internal systems audits or evaluations are conducted with an opening meeting in which representatives from management, key operational staff, and QA staff participate. The opening meeting provides a review of the objectives of the audit and the schedule required to conduct the audit. At the completion of the audit, a debriefing is held to outline the findings, including identification of positive performance, to discuss requirements in areas of deficiencies, and to answer questions. Spot assessments a

generally more informal than systems audits, and may be conducted without prior scheduling.

Internal systems audits or evaluations are conducted at a minimum yearly by QA staff external to the lab. Spot assessments are conducted per schedule by the laboratory QA group.

The findings of all audits and assessments are documented as is the laboratory response and any corrective actions. Follow-up checks are performed and the status of implementation of corrective actions is documented for all categories of audits and assessments. This cycle continues until all issues are closed.

9.2.1 Performance Audits

Performance audits or performance evaluations (PEs) are conducted to verify the ability of the laboratory to correctly identify and quantitate compounds in PE samples. These PE samples may be supplied internally or externally as single-blind or double-blind samples and can be used to assess if a deficiency has been corrected. The results of internal performance audits may be used to document the proficiency of the analyst performing the work or to assess the overall performance of an analytical method. Double-blind performance audits are conducted by Quanterra® Corporate QA to assess the effectiveness of all aspects of laboratory operation from project initiation through analysis and reporting. In addition, any problems detected regarding quotes, bids, and invoicing are brought to the attention of the PM and to the finance/accounting department, as appropriate.

The results of each performance audit shall be reported to laboratory management. All performance audit results which are identified as unacceptable must be investigated. The findings of the investigation and corrective action taken must be documented.

9.2.2 Systems Audits

A systems audit assesses fulfillment of the QAMP and the effectiveness of the quality system. Each laboratory undergoes numerous systems audits performed by external parties, including federal, state, and local regulatory authorities and clients.

9.2.2.1 Independent Internal Systems Audits or Evaluations

Each year, an independent systems evaluation will be performed under the direction of the Corporate Director of Quality Assurance and according to Corporate SOP Number, CORP-QA-0014. This evaluation is performed to assess each laboratory's adherence to the requirements of the QAMP, SOPs, internal policies, and to assess the status of corrective actions from other audits at that facility.

The Corporate Director of Quality Assurance shall appoint a lead auditor to conduct the systems evaluation. An auditor must be a Quanterra Quality Assurance Manager with direct experience on at least one Quanterra systems audit or a certified Quanterra Lead Auditor as defined in the SOP, CORP-QA-0013, Employee Orientation and Training. A corporate audit outline shall be used. The lead auditor has the authority to lengthen the evaluation, revise the scope of the evaluation, stop work, or specify an accelerated schedule for re-evaluation. The lead auditor shall be responsible for preparing a report detailing the results of the evaluation. The report shall be submitted, after approval by Corporate QA, to the audited Laboratory Manager, Regional General Manager, and Laboratory QA Manager within five weeks of the audit. A copy of the report shall be distributed to the Corporate QA Director. The audit report shall provide a summary of the audit results with the auditor's comments and the findings that were determined by the lead auditor.

The evaluated laboratory must respond to Corporate QA in writing within four weeks of receiving the evaluation report. The QA Manager is responsible for coordinating the response to the evaluation report. The Laboratory Manager or Operations Manager must approve all responses to internal evaluation reports prior to submittal to the auditor.

The evaluation may result in findings, areas needing improvement, and notable practices. Each term will be defined as such:

- Findings - are defined as those noncompliant practices or policies which have significant adverse impact on data quality, technical defensibility, or regulatory acceptance of data. Findings require immediate attention by the laboratory management and must be resolved to comply with Quanterra®'s quality documents and laboratory-established procedures.

- Areas Needing Improvement - represent isolated instances of noncompliance or issues that are judged to have a less immediate impact on data quality. Laboratory management must correct the situation or otherwise ensure that the condition does not recur.

- Notable Practices - should also be pointed out during the audit. These are defined as laboratory practices that increase effectiveness and quality and represent improvements with respect to conventional laboratory operations.

Operations management is responsible for taking corrective action to address the findings and areas needing improvement (deficiencies) identified during the audit.

It is the responsibility of the QA Manager at each facility to verify implementation of the corrective actions and document the closure of all internal evaluation findings. This process shall be documented and provided to the recipients of the original audit report. This process is further described in the Quanterra® Corporate SOP Number CORP-QA-0014, "Laboratory Internal Systems Evaluation".

Internal evaluation reports shall be maintained according to the Quanterra® Record Retention Policy (LEG-004) as confidential documents and shall not be released for use outside the laboratory. External auditors may view internal audit reports as part of their on-site audit.

9.2.2.2 External Systems Audits

Audits of Quanterra® laboratories are performed by external agencies and clients. All scheduled audits shall be placed on the facility's calendar with the knowledge of the Laboratory Manager and the Laboratory QA Manager to assure no scheduling conflicts occur and that appropriate staff will be available to meet the agencies' or clients' objectives.

All deficiencies reported to the laboratory must be responded to within the time line specified by the client. A plan of action to correct the deficiencies, as well as corrective actions taken, must be documented. It is the responsibility of the QA Manager to coordinate the response to the audit report. The development and implementation of the corrective actions is the responsibility of the Operations management as related to their respective areas. All responses must be approved by the Laboratory Manager or Operations Manager prior to submittal to the auditor. A copy of the audit report and the laboratory's response must be provided to the Laboratory Manager. It is the responsibility of the QA Manager to verify implementation of the corrective actions and inform the responsible manager of the closure of all deficiencies from the audit.

9.2.3 Data Audits

Data audits are routinely performed and documented to ensure that project records meet project requirements as described in method SOPs, project plans, or other documented requirements. The data audit is used to identify any lab errors that may have occurred. The laboratory QA Manager or designee is responsible for performing data audits as specified in Quanterra® SOP Number CORP-QA-0004, "QA Data Review". This independent review may also serve as the annual assessment of analysts in verifying initial and/or on-going technical capability of chemists performing routine analyses.

9.2.4 Spot Assessments

Spot assessments are conducted to monitor or observe a process or activity in order to verify

conformance to the SOP requirements for that activity. The frequency, normally monthly, for performing these assessments is determined by the facility QA Manager. The scope of the assessment is also determined by the QA Manager and may be directed based on information obtained from client inquiries, trends in recorded nonconformances, performance audits, or other sources. A spot assessment may be used to assess a procedure performance relative to the documented SOP. This assessment identifies deviations from requirements that may not be detected in a detailed review of the data package alone. Such an assessment is conducted by observation of the associates performing the task compared with the documented SOP. In some cases, the assessment may be conducted through interviews with the associate when observation of a task is not possible. Review of relevant documentation for the completed procedure is included in such an assessment. A checklist may be used in conducting the assessment. The results of the assessment are documented, as are the corrective actions. All deficiencies noted as a result of a spot assessment must be corrected by the responsible staff in a timely manner.

9.3 Client Inquiries and Complaints

Client inquiries and complaints are generally received through the PM or a member of the CST. Typically, the PM communicates with the client to determine the details of the inquiries, including technical data problems, deliverable issues, turn-around-time problems, etc. Technical and deliverable issues are coordinated by the PM and usually involve input from operations, QA, and management staff. A formal written response to the client is coordinated by the PM, but may on occasion be delivered by the CSM or the Account Manager. Details of the types and levels of complaints and required documentation are provided in Corporate Policy No. QA-013, "Procedures to Address Customer Complaints".

9.4 Quality Reports to Management

The QA Manager and Corporate Director of Quality Assurance shall prepare and maintain copies of reports to management on a monthly basis indicating the effectiveness of the QMS. The operations monthly QA reports to management shall, at a minimum, include discussion of the following activities that occurred during the month:

- Internal/external audit findings
- Certification changes
- External/internal PE sample results
- Summary of client inquiries for which QA was asked to assist the laboratory in developing a response
- Data quality investigations.

- NCM/lab issues

The corporate reports serve as executive summaries to members of senior management of the detailed information provided by the QA Managers.

9.5 Management Process Review

Based upon laboratory audits or input from clients or the QA group, management may elect to assess any of the operations or processes therein. These reviews and corrective actions would be documented and followed up by management. The purpose of these assessments would be to correct systematic issues or processes before they impact data quality.

9.6 Management Review of the QMS

Annually laboratory management will evaluate the status of the quality systems in the laboratory to ensure the procedures and policies are in place and they are adhered to. The Corporate Director of Quality Assurance annually sends to the laboratory management an outline of the Quality Systems with a request that each operation reviews their status including completion dates on any items requiring correction or implementation. The laboratory management report their assessments with any recommendations for changes or improvements to the Corporate QA Director which in turn will report results to the Chief Operating Officer.

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10.0 Quality Improvement

Quality improvement at Quanterra® is a critical element of our quality strategy as well as our business strategy. Quanterra® will become a world class organization through a commitment to continuous quality improvement. Every Quanterra® associate must understand that continuous improvement is a guiding principle pertinent to all aspects of our business.

The key elements of quality improvement are:

- Standardization of procedures
- Continuous quality improvement
- Benchmarking
- Quality assessment
- Understanding the clients' needs
- Quality measures and standards.

10.1 Standardization of Procedures

Due to the vast number of methods that have been introduced into the environmental analytical field, as well as revisions and proposed updates to currently promulgated methods, many methodologies contain conflicting requirements. It is through the generation and use of standard operating procedures, which contain Quanterra®'s best technical interpretation of published methods, that Quanterra® succeeds in providing quality standardized analytical testing to our clients. This standardization is achieved through the implementation of standardized or corporate SOPs and policies. Quanterra® will generate and implement new SOPs and policies as the need arises, and will train associates in their importance and use.

The standardization of procedures, as they are implemented throughout Quanterra®, result in improved quality of our services and deliverables provided to our clients at some level at all operations.

Any departures from the standardized procedures must be documented and, if a deficiency is noted, it is documented with a NCM. For further details refer to Section 9.1.1.

10.2 Continuous Quality Improvement

The continuous improvement of processes throughout Quanterra® is the responsibility of each Quanterra® associate. Processes are followed in every operating unit, group, and by each individual, and each of these processes is applicable to this discussion. All associates are empowered and encouraged to bring suggestions for process improvement

changes to the attention of laboratory management, as well as the Technology and QA groups. Management is responsible for assessing the recommendation for improvement and following through with an implementation plan when appropriate.

10.3 Benchmarking

Benchmarking is a step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices. Benchmarking is performed within Quanterra®, as well as on the industry. Changes to processes resulting from benchmarking may be tested in a single laboratory under senior technical and QA management. If successful, the process change may be implemented throughout Quanterra®.

10.4 Quality Assessment

Quality improvement is also achieved through the practice of quality assessment and subsequent correction of detected quality-related problems when they occur. Quality assessment and response are described in Section 9.0.

10.5 Understanding the Client's Needs

Client satisfaction must be a goal of all Quanterra® associates. Periodic client surveys provide a useful tool to measure if we are meeting their needs. While quality is defined as meeting the requirements of our clients, both internal and external, we must also strive to exceed our client's expectations. Every Quanterra® associate is either directly or indirectly involved in meeting our client's needs, therefore effective communication of these needs to all associates as described in this QAMP is essential.

Any verbal or written inquires or complaints from clients to any associate must be addressed in a timely manner and any issue concerning data quality must be documented. (See Section 9.3).

10.6 Quality Measures and Standards

Measures and standards used by Quanterra® are fundamental to assessing and achieving our commitment to continuous improvement. With most business processes, performance is generally measured at the end. Quanterra® managers must provide continuous feedback to all associates regarding performance measures and standards.

Key Result Indicators (KRIs) provide one of Quanterra®'s measures. Additional measures and standards shall be added to track programs, processes, or projects, as appropriate.

KRIs measure performance in areas considered critical to achieving world class Quality in customer satisfaction and business performance. KRIs focus quality measures on the customer as well as the processes that our customers value. KRIs are intended to demonstrate continuous improvement and focus on the "vital few" issues for the business. Quanterra® KRIs include:

- On-time delivery
- Holding time violations
- Reissued reports
- Turn-around-time
- Safety.

Measurements of performance include internal and external audits, performance evaluations, and double-blind evaluations which are further described in Section 9.0. Improved results from these measurements show successfulness of process improvements.

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Tables

Quanterra®
Quality Assurance Management Plan

Table Section

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Table of Contents

Table No.	Title	Page
2.3-1	Quanterra® Quality Assurance Management Plan Requirements Matrix	5
2.3-2	Cross-Reference of QAMP Sections Addressing NELAC Quality Manual Requirements	9
4.2-1	List of Quanterra® Quality-Related Items that Require Evaluation Prior to Use	12
5.1-1	Quanterra® Quality Documents and Required Approval	13
5.2-1	Quanterra® Quality Document Review Requirements	14
8.4-1	Field Quality Control Samples	15
8.4-2	Laboratory Quality Control Samples	16
8.4-3	Laboratory Performance Quality Control Samples	17
8.4-4	Matrix Specific Quality Control Samples	17
8.4-5	Inorganic Laboratory Quality Control Samples	18
8.4-6	Organic Laboratory Quality Control Samples	67
8.4-7	USEPA Contract Laboratory Program Statement of Work Quality Control Samples	96
8.5-1	Inorganic Sample Containers, Preservatives, and Holding Times	110
8.5-2	Organic Sample Containers, Preservatives, and Holding Times	122
8.5-3	Radiological Sample Containers, Preservatives, and Holding Times	136
8.5-4	Sample Containers, Preservatives, and Holding Times for USEPA Contract Laboratory Program Statement of Work	139
8.5-5	Sample Containers, Preservatives, and Holding Times for TCLP and SPLP	141
8.5-6	Periodic Equipment Calibrations	142
8.5-7	Summary of Inorganic Method Calibrations	143
8.5-8	Summary of Organic Method Calibrations	159
8.5-9	Summary of USEPA Contract Laboratory Program Statement of Work Method Calibrations	169
8.6-1	Precision and Accuracy Measurements	173
8.11-1	Instrument Maintenance Schedule - Ion Chromatograph	175
8.11-2	Instrument Maintenance Schedule - LACHAT Auto Analyzer	175
8.11-3	Instrument Maintenance Schedule - Total Organic Halide Analyzer	176
8.11-4	Instrument Maintenance Schedule - High Pressure Liquid Chromatograph	176
8.11-5	Instrument Maintenance Schedule - Flame Atomic Absorption Spectroscopy	177
8.11-6	Instrument Maintenance Schedule - Inductively Coupled Argon Plasma/ Mass Spectrometry (ICAP/MS)	177
8.11-7	Instrument Maintenance Schedule - ICP	178
8.11-8	Instrument Maintenance Schedule - Graphite Furnace Atomic Absorption	179
8.11-9	Instrument Maintenance Schedule - Cold Vapor Atomic Absorption (Leeman PS 200)	179

Table of Contents (continued)

Table No.	Title	Page
8.11-10	Instrument Maintenance Schedule - Cold Vapor Atomic Absorption (PE 5000)	180
8.11-11	Instrument Maintenance Schedule - Gas Chromatograph	181
8.11-12	Instrument Maintenance Schedule - Mass Spectrometer	183
8.11-13	Instrument Maintenance Schedule - TRAACS 800 Auto Analyzer	184
8.11-14	Instrument Maintenance Schedule - Sonicator	184
8.11-15	Instrument Maintenance Schedule - Analytical/Top Loading Balances	184
8.11-16	Instrument Maintenance Schedule - Refrigerators/Walk-in Coolers	184
8.11-17	Instrument Maintenance Schedule - Ovens	185
8.11-18	Instrument Maintenance Schedule - Specific Digital Ion Analyzer	185
8.11-19	Instrument Maintenance Schedule - Turbidimeter	185
8.11-20	Instrument Maintenance Schedule - Dissolved Oxygen Meter	185
8.11-21	Instrument Maintenance Schedule - Conductance Meter	186
8.11-22	Instrument Maintenance Schedule - Chemical Oxygen Demand (COD) Reactor	186
8.11-23	Instrument Maintenance Schedule - Spectrophotometer	186
8.11-24	Instrument Maintenance Schedule - pH Meter	187
8.11-25	Instrument Maintenance Schedule - Fourier Transform Infrared Spectrometry	187
8.11-26	Instrument Maintenance Schedule - Radiological Analysis Equipment	188
8.11-27	Instrument Maintenance Schedule - Total Organic Carbon Analyzer (OI 7000)	189
8.11-28	Instrument Maintenance Schedule - APCI/ESI LC/MS/MS	190
8.11-29	Instrument Maintenance Schedule -Digestion Block	191
8.11-30	Instrument Maintenance Schedule - Flash Point Tester	191

TABLE 2.3-1

Quanterra® Quality Assurance Management Plan Requirements Matrix

EPA QA/R-2	Quanterra QAMP (Rev 3)	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
1 Management and Organization	1.0 Management Commitment and Organization	2.1 Management and Organization	1 Organization	9.a. General	1.1 Introduction 1.2 Purpose 1.3 Scope	5.0 Management Responsibility
2 Quality System and Description	2.0 Quality System and Description	2.2 Quality System and Description	2 Quality Assurance Program	1 Program	2.1 Special Word Usage 2.2 Specific Terms 5.1 Quality Assurance 5.2 Quality Control	5.2 Quality System
3 Personnel Qualification and Training	3.0 Associate Qualification and Training	2.3 Personnel Training and Qualification	2 Quality Assurance Program	2 Personnel Training and Qualification	3.2 Personnel Preparation	14.0 Personnel
4 Procurement of Items and Services	4.0 Procurement of Items and Services	2.4 Procurement of Items and Services	4 Procurement Document Control 7 Control of Purchased Items and Services	7 Procurement	N/A	7.0 Quality in Procurement 13.0 Subcontracting
5 Documentation and Records	5.0 Documentation and Records	2.5 Documents and Records	6 Document Control 17 Quality Assurance Records	4 Documents and Records	3.6 Direct Bioassay-Record Retention 4.5 Indirect Bioassay-Record Retention	8.4 Quality Documentation and Records
6 Computer Hardware and Software	6.0 Computer Hardware and Software	2.6 Computer Hardware and Software	3 Design Control 11 Test Control	N/A	N/A	ISO 9000-3[4]

TABLE 2.3-1

Quanterra® Quality Assurance Management Plan Requirements Matrix (Continued)

EPA QA/R-2	Quanterra QAMP (Rev 3)	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
7 Planning	7.0 • Planning	2.7 Planning 3.1 Planning and Scoping 3.3 Implementation of Planned Operations	2 Quality Assurance Program 3 Design Control 5 Instructions, Procedures, and Drawings 8 Identification and Control of Items 9 Control of Processes 11 Test Control 13 Handling, Storage, and Shipping	1 Program 6 Design N/A	3.1 Facility Criteria 3.4 Direct Bioassay-Performance Criteria for Service Laboratories 3.5 Direct Bioassay-Reporting Results 4.1 Indirect Bioassay-Responsibilities of the Service Laboratory Customer 4.2 Indirect Bioassay-Analytical Methodology 4.3 Indirect Bioassay-Performance Criteria for Service Laboratories 5.2 Quality Control	6.3.3 Quality Plans
8 Implementation of Work Processes	8.0 Work Processes and Operations	2.8 Implementation of Work Processes	1 Organization 5 Instructions, Procedures, and Drawings	5 Work Processes 6 Design	3.1 Facility Criteria	8.0 Laboratory Operations Quality Assurance 9.0 Control of Measuring and Test Equipment 10.0 Data Validation 15.0 Use of Statistical Methods

TABLE 2.3-1
Quanterra® Quality Assurance Management Plan Requirements Matrix
(Continued)

EPA QA/R-2	Quanterra QAMP (Rev 3)	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
8 Implementation of Work Processes (Continued)		3.2 Design of Data Collection Operations	10 Inspection 12 Control of Measuring and Test Equipment 14 Inspection, Test, and Operating Status	8 Inspection and Acceptance Testing		
9 Assessment and Response[3]	9.0 Quality Assessment and Response	2.9 Assessment and Response 3.4 Assessment and Response 3.5 Assessment and Verification of Data Usability	2 Quality Assurance Program 13 Handling, Storage, and Shipping 15 Control of Non-conforming Items 16 Corrective Action 18 Audits	9 Management Assessment 10 Independent Assessment	3.3 Direct Bioassay-Interpretation of Measurements 3.5 Direct Bioassay-Reporting Results 4.4 Indirect Bioassay-Reporting Results 6.1 Direct Bioassay Measurements 6.2 Indirect Bioassay Measurements	16.0 Nonconformity 17.0 Corrective Action 18.0 Auditing the Quality System
9 Quality Improvement[3]	10.0 Quality Improvement	2.10 Quality Improvement	N/A	3 Quality Improvement	N/A	N/A

TABLE 2.3-1
Quanterra® Quality Assurance Management Plan Requirements Matrix
(Continued)

Footnotes

(1) Section II, "Basic Requirements."
(2) Criterion from Section 9, "Requirements."
(3) Technically equivalent to ISO 9001.
(4) Quality Management and Quality Assurance Standards, ISO 9000, Part 3, "Guidelines for the Application of ISO 9001 to the Development, Supply and Maintenance of Software."
(5) This document has two sections numbered "9."

TABLE 2.3-2

CROSS-REFERENCE OF QAMP SECTIONS ADDRESSING NELAC QUALITY MANUAL REQUIREMENTS

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	QUANTERRA® QAMP REVISION 3, QA SOP OR POLICY REFERENCE
a) A quality policy statement, including objectives and commitments, by top management	QAMP Chapter 1
b) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts	QAMP Chapter 1
c) The relationship between management, technical operations, support services and the quality system	QAMP Chapter 1 QAMP Chapter 7
d) Procedures to ensure that all records required under this chapter are retained as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force	QAMP Chapter 2 QAMP Chapter 5
e) Job descriptions of key staff and reference to the job descriptions of other staff	QAMP Chapter 1 and Chapter 3 Separate document of job descriptions available from Lab-HR Manual
f) Identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed concurrence, (with appropriate titles) of all responsible parties including the QA officer, technical director, and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager	QAMP Title/Approval Page Facility Specific Appendix Approval Page
g) The laboratory's procedures for achieving traceability of measurements	QAMP Chapter 8

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, July 2, 1998

TABLE 2.3-2

CROSS-REFERENCE OF QAMP SECTIONS ADDRESSING
NELAC QUALITY MANUAL REQUIREMENTS
(continued)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	QUANTERRA® QAMP REVISION 3, QA SOP OR POLICY REFERENCE
a) A list of all test methods under which the laboratory performs its accredited testing	Facility Specific Appendices
b) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	QAMP Chapter 7
c) Reference to the calibration and/or verification test procedures used	QAMP Chapter 8 Table 8-4,5,6,7
d) Procedures for handling submitted samples	QAMP Chapter 8
e) Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests	QAMP Chapter 8 Facility Specific Appendices
f) Reference to procedures for calibration, verification and maintenance of equipment	QAMP Chapter 8
g) Reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes	QAMP Chapter 9
h) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	QAMP Chapter 9 and 10 SOP CORP-QA-0010, "Nonconformance and Corrective Action"

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, July 2, 1998

TABLE 2.3-2

CROSS-REFERENCE OF QAMP SECTIONS ADDRESSING
NELAC QUALITY MANUAL REQUIREMENTS
(continued)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	QUANTERRA® QAMP REVISION 3, QA SOP OR POLICY REFERENCE
j) The laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	QAMP Chapter 9
k) Procedures for dealing with complaints	QAMP Chapter 9 Policy QA-013 "Procedures to Address Customer Complaints"
l) Procedures for protecting confidentiality and proprietary rights (including national security concerns)	QAMP Chapter 5 Corporate Legal Policy
m) Procedures for audits and data reviews	QAMP Chapter 8 QAMP Chapter 8 and 9 Policy QA-012, "Technical Data Review Requirements" SOP CORP-QA-0004 "Independent QA Data Review"
n) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and/or receive any needed training	QAMP Chapter 3 SOP CORP-QA-0013, "Employee Orientation and Training"
o) Reference to procedures for reporting analytical results	QAMP Chapter 8 Policy QA-009 "Establishment of Reporting Limits"
p) A table of contents and applicable list of references, glossaries, and appendices	QAMP Table of Contents List of QA Policies and SOPs Table 2.3-2

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, July 2, 1998

TABLE 4.2-1

List of Quanterra® Quality-Related Items that Require Evaluation Prior to Use

Quality-Related Item	Standard Operating Procedure for Quality Testing
Acetone	CORP-QA-0001
Dichloromethane	CORP-QA-0001
Hexane	CORP-QA-0001
Hydrochloric acid	CORP-QA-0001
Freon	CORP-QA-0001
Methanol	CORP-QA-0001
Nitric acid	CORP-QA-0001
Hydrogen Peroxide	CORP-QA-0001
Sulfuric acid	CORP-QA-0001
Toluene	CORP-QA-0001

TABLE 5.1-1
Quanterra® Quality Documents and Required Approval

Quality Document	Required Approvals
Quality Assurance Management Plan (QAMP)	• Corporate Director of Quality Assurance • Vice President of Operations Services • Chief Operating Officer
Quality Assurance Management Plan (QAMP) Facility Appendix	• Quality Assurance Manager • Technical Manager • Laboratory Manager • Regional General Manager/Laboratory Director • Corporate Director of Quality Assurance
Corporate Standard Operating Procedures (SOPs)	• Corporate Director of Quality Assurance • Corporate Director of Environmental Health and Safety[1] • Management (generally a Vice President) • Technical Specialist (technical SOPs only)
Operation-Specific Standard Operating Procedures (SOPs)	• Quality Assurance Manager • Laboratory Health and Safety Coordinator[1] • Laboratory Manager • Technical Specialist
Quality Policy Documents	• Corporate Director of Quality Assurance • Vice President of Operations Services

[1] Required only if procedure encompasses more than standard office safety requirements.

TABLE 5.2-1
Quanterra® Quality Document Review Requirements

Document Type	Frequency of Review	Responsible Party
Quality Assurance Management Plan (QAMP)	Every Two Years	Corporate Director of Quality Assurance
Quality Assurance Management Plan (QAMP) Facility Appendix	Annual	Quality Assurance Manager
Corporate Standard Operating Procedures (SOP)	Every Two Years	Corporate Technology/QA
Operation-Specific Standard Operating Procedures (SOP)	Every Two Years	Laboratory Staff
Quality Policy Documents	Every Two Years	Corporate Director of Quality Assurance

TABLE 8.4-1
Field Quality Control Samples

Type	Applicability		Accuracy and Precision Application	Introduced By
	Inorganic	Organic		
Trip Blank (volatiles)	No	Yes	Accuracy	Supplier of Containers
Field Blank	Yes	Yes	Accuracy	Field Sampler
Rinsate Blank	Yes	Yes	Accuracy	Field Sampler
Collocated Sample	Yes	Yes	Precision	Field Sampler
Split Sample	Yes	Yes	Precision	Field Sampler
Field Duplicate	Yes	Yes	Precision	Field Sampler
Field Matrix Spike	Yes	Yes	Accuracy	Field Sampler

TABLE 8.4-2
Laboratory Quality Control Samples

Type	Frequency	Applicability		Accuracy and Precision Application	Introduced By
		Inorganic/ Radiochemical	Organic		
Analytical Spike	As specified in methods, or as needed	Yes	No	Accuracy	Analyst/ Prep
Duplicate	1 out of 20 or at least 1/month/run	Yes	Yes	Precision	Analyst/ Prep
Instrument Blank	As specified methods, or as needed	Yes	Yes	Accuracy	Analyst
Interference Check Sample	As specified in methods	Yes	No	Accuracy	Analyst
Internal Standard	Each sample and standard	Yes	Yes	Both	Analyst/ Prep
Laboratory Control Sample	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike Duplicate	1 per each group of samples processed up to 20 samples.	Yes	Yes	Both	Analyst/ Prep
Method Blank	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Surrogate	All standards, method blanks, LCS, and samples.	No	Yes Method Dependent	Accuracy	Analyst/ Prep
Yield Monitor	Operation-specific	Yes	No	Accuracy	Prep

TABLE 8.4-3
Laboratory Performance Quality Control Samples

Sample/Measurement	Purpose
Method Blanks	Demonstrates that the laboratory systems (*e.g.*, glassware cleaning procedures) and laboratory reagents used for the preparation and analysis of samples have not contributed to a false positive or negative measurement.
Instrument Blank	Demonstrates that the analytical system has not contributed to a false positive or negative measurement.
Laboratory Control Sample	Demonstrates the laboratory's ability to perform an analysis within the performance requirements of the method.

TABLE 8.4-4
Matrix Specific Quality Control Samples

Quality Control Sample	Purpose
Duplicate Samples	Estimates the ability of the laboratory to obtain precise measurements on a sample. This measure is dependent on the homogeneity of the sample being duplicated. Solid samples often portray poor sample homogeneity and therefore often have poor duplication with regards to the sample result.
Matrix Spike Sample	Estimates the ability of the laboratory to obtain accurate measurements on a sample. The measure is dependent on the bias a sample matrix may cause regarding a given analyte.
Matrix Spike Duplicate Sample	In addition to verifying the accuracy of the matrix spike sample, the matrix spike duplicate can be used with the matrix spike sample as a measure of precision by calculating the relative percent difference (RPD).

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Acidity	Method Blank	305.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	305.1	Not Applicable	—	Not Applicable
	Matrix Spike	305.1	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	305.1	Not Applicable	—	Not Applicable
	Duplicate	305.1	Frequency: 1 per batch of 10 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Alkalinity	Method Blank	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	310.1 2320B	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	310.1 2320B	Not Applicable	—	Not Applicable
	Duplicate	310.1 2320B	Frequency: 1 per batch of 10 samples Criteria 310.1: ≤ 20 % RPD[3] Criteria 2320B: ≤ 25 % RPD[3] Corrective Action: Flag data outside of limit.	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Ammonia	Method Blank	350.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	350.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	350.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	350.1	Not Applicable	—	Not Applicable
	Duplicate	350.1	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Ammonia (TKN)	Method Blank	351.2 351.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	351.2 351.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	351.2 351.3	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	351.2 351.3	Not Applicable	—	Not Applicable
	Duplicate	351.2 351.3	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
BOD	Method Blank	405.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	405.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	405.1	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	405.1	Not Applicable	—	Not Applicable
	Duplicate	405.1	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Bromide	Method Blank	300.0[5] 320.1 D1246	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5] 320.1 D1246	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Matrix Spike	300.0[5] 320.1 D1246	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0[5] 320.1 D1246	Not Applicable	9056	Not Applicable
	Duplicate	300.0[5] 320.1 D1246	Methods 300.0, 320.1: Not Applicable Frequency: Method D1246: 1 with each batch of samples processed not to exceed 20 samples	9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chemical Oxygen Demand (COD)	Method Blank	410.1 410.2 410.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	410.1 410.2 410.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	410.1 410.2 410.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	410.1 410.2 410.4	Not Applicable	—	Not Applicable
	Duplicate	410.1 410.2 410.4	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chloride	Method Blank	300.0[5] 325.1 325.2 325.3 4500-Cl E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056 9251 9253	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5] 325.1 325.2 325.3 4500-Cl E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056 9251 9253	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0[5] 325.1 325.2 325.3 4500-Cl E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056 9251 9253	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Methods 9251Corrective Action: If not within laboratory control limits, rerun all associated samples Method 9056/9253 Corrective Action: Flag data associated with MS outside of limits

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chloride (continued)	Matrix Spike Duplicate	300.0[5] 325.1 325.2 325.3 4500-Cl E	Not Applicable	9056 9251 9253	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits/< 20 % RPD[3] Corrective Action: Flag data outside of limit Method 9056: MSD is not applicable
	Duplicate	300.0[5] 325.1 325.2 325.3 4500-Cl E	Methods 300.0, 325.1, 325.2, 325.3: Not Applicable Method 4500-Cl E: Frequency: 1 with each batch of samples processed not to exceed 20 samples	9056 9251 9253	Method 9056/9253: Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Chlorine, Residual	Method Blank	330.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chlorine, Residual (continued)	Laboratory Control Sample	330.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	330.1 330.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	330.1 330.3	Not Applicable	—	Not Applicable
	Duplicate	330.1 330.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	—	Water

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chromium (Cr^{+6})	Method Blank	218.4 3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	218.4 3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples prepped Criteria: percent recovery for water must be within ± 15 % and for solids must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	218.4 3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	218.4 3500 Cr-D	Not Applicable	3060A 7196A	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chromium (Cr^{+6}) (continued)	Duplicate	218.4 3500 Cr-D	Not Applicable	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] limit Corrective Action: Flag data outside of limit.
Color	Method Blank	110.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	110.2	Not Applicable	—	Not Applicable
	Matrix Spike	110.2	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	110.2	Not Applicable	—	Not Applicable
	Duplicate	110.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	—	Not Applicable
Conductivity	Method Blank	120.1	Not Applicable	9050A	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Conductivity (continued)	Laboratory Control Sample	120.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1	Not Applicable	9050A	Not Applicable
	Matrix Spike Duplicate	120.1	Not Applicable	9050A	Not Applicable
	Duplicate	120.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	9050A	Frequency: 1 with each batch of samples processed not to exceed 10 samples
Cyanide (Amenable)	Method Blank	335.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Cyanide (Amenable) (continued)	Laboratory Control Sample	335.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	335.1	Not Applicable	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.1	Not Applicable	9010B 9012A	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Cyanide (Total)	Method Blank	335.2 335.3 4500-CN E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.2 335.3 4500-CN E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 4500-CN E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limit is 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Cyanide (Total) (continued)	Matrix Spike Duplicate	335.2 335.3 4500-CN E	Not Applicable	9010B 9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limit is 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.2 335.3	Methods 335.2, 335.3: Not Applicable Method 4500-CN E: Frequecny: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	9010B 9012A	Not Applicable
Flashpoint	Method Blank	—	Not Applicable	1010 1020A	Not Applicable
	Laboratory Control Sample	—	Not Applicable	1010 1020A	Not Applicable
	Matrix Spike	—	Not Applicable	1010 1020A	Not Applicable
	Matrix Spike Duplicate	—	Not Applicable	1010 1020A	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Flashpoint (continued)	Duplicate	—	Not Applicable	1010 1020A	Frequency: 1 per batch of ≤20 samples Criteria: RPD[3] must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate
Fluoride	Method Blank	300.0[5] 340.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable
	Laboratory Control Sample	300.0[5] 340.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Fluoride (continued)	Matrix Spike	300.0[5] 340.2	Frequency: 1 per 10 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with outside of limit
	Matrix Spike Duplicate	300.0[5] 340.2	Not Applicable	9056	Not Applicable
	Duplicate	300.0[5] 340.2	Not Applicable	9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Hardness	Method Blank	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Hardness (continued)	Laboratory Control Sample	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	130.2 2340B	Method 130.2, Not Applicable Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	Not Applicable
	Matrix Spike Duplicate	130.2 2340B	Method 130.2, Not Applicable Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	Not Applicable
	Duplicate	130.2 2340B	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Iodide	Method Blank	345.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	345.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	345.1	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	345.1	Not Applicable	—	Not Applicable
	Duplicate	345.1	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Iron	Method Blank	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	3500-Fe D	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Iron (continued)	Duplicate	3500-Fe D	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	Not Applicable
Methylene Blue Active Substances (MBAS)	Method Blank	425.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	425.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Methylene Blue Active Substances (MBAS) (continued)	Matrix Spike	425.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	425.1	Not Applicable	—	Not Applicable
	Duplicate	425.1	Not Applicable	—	Not Applicable
Nitrate	Method Blank	300.0[5] 352.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056 9210	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Criteria 9210: Concentration must be < 1 mg/L of Nitrate Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5] 352.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056 9210	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Criteria 9210: 90-110% Corrective Action: If not within laboratory control limits, rerun all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrate (continued)	Matrix Spike	300.0[5] 352.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056 9210	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Criteria 9210: 75-125% Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0[5] 352.1	Not Applicable	9056 9210	Not applicable
	Duplicate	300.0[5] 352.1	Not Applicable	9056 9210	Method 9056: Frequency: 1 per 10 samples Criteria: RPD[3] must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples Method 9210: Not applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrite	Method Blank	300.0[5] 354.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5] 354.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0[5] 354.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0[5] 354.1	Not Applicable	9056	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrite (continued)	Duplicate	300.0[5] 354.1	Not Applicable	9056	Frequency: 1 per 10 samples Criteria: RPD[3] must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
Nitrate-Nitrite	Method Blank	353.1 353.2 353.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	353.1 353.2 353.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrate-Nitrite (continued)	Matrix Spike	353.1 353.2 353.3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	353.1 353.2 353.3	Not Applicable	—	Not Applicable
	Duplicate	353.1 353.2 353.3	Not Applicable	—	Not Applicable
Odor	Method Blank	140.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	140.1	Not Applicable	—	Not Applicable
	Matrix Spike	140.1	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	140.1	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Odor (continued)	Duplicate	140.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	—	Not Applicable
pH	Method Blank	150.1 4500-H^+ B	Not Applicable	9040B 9045C	Not Applicable
	Laboratory Control Sample	150.1 4500-H^+ B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Sample provided by external source, must be within ± 0.05 pH units Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Sample provided by external source, must be within ± 0.05 pH units Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	150.1 4500-H^+ B	Not Applicable	9040B 9045C	Not Applicable
	Matrix Spike Duplicate	150.1 4500-H^+ B	Not Applicable	9040B 9045C	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
pH (continued)	Duplicate	150.1 4500-H^+ B	Method 150.1 Frequency: 1 with each batch of samples processed not to exceed 20 samples Method 4500-H^+ B Frequency: 1 with each batch of 10 samples Method 150.1 Criteria: ≤ 20 % RPD[3] limit Method 4500-H^+ B Criteria: ≤ 25 % RPD[3] limit Corrective Action: Flag data outside of limit.	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are ≤ 20% RPD[3] Corrective Action: Flag data associated with unacceptable Duplicate
Phenolics	Method Blank	420.1 420.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	420.1 420.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Phenolics (continued)	Matrix Spike	420.1 420.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	420.1 420.2	Not Applicable	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Duplicate	420.1 420.2	Not Applicable	9065 9066	Not Applicable
Phosphate	Method Blank	---	Not Applicable	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	---	Not Applicable	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Phosphate (continued)	Matrix Spike	—	Not Applicable	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	—	Not Applicable	9056	Not Applicable
	Duplicate	—	Not Applicable	9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Phosphorus (Total and Ortho-phosphate)	Method Blank	300.0[4,5] 365.1 365.2 365.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Phosphorus (Total and Ortho-phosphate) (continued)	Laboratory Control Sample	300.0[4,5] 365.1 365.2 365.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	300.0[4,5] 365.1 365.2 365.3	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	300.0[4,5] 365.1 365.2 365.3	Not Applicable	—	Not Applicable
	Duplicate	300.0[4,5] 365.1 365.2 365.3	Not Applicable	—	Not Applicable
Reactivity (Cyanide and Sulfide)	Method Blank	---	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Reactivity (Cyanide and Sulfide) (continued)	Laboratory Control Sample	—	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	—	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Follow QC sample requirements of determinative method
	Matrix Spike Duplicate	—	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Follow QC sample requirements of determinative method
	Duplicate	—	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Not Applicable
Silica, Dissolved	Method Blank	370.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Silica, Dissolved (continued)	Laboratory Control Sample	370.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	370.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	370.1	Not Applicable	—	Not Applicable
	Duplicate	370.1	Not Applicable	—	Not Applicable
Solids	Method Blank	160.1 160.2 160.3 160.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is ≥ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed. SOP No. CORP-WC-0002.	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Solids (continued)	Laboratory Control Sample	160.1 160.2 160.3 160.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, reprepare and rerun all associated samples	—	Not Applicable
	Matrix Spike	160.1 160.2 160.3 160.4	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4	Not Applicable	—	Not Applicable
	Duplicate	160.1 160.2 160.3 160.4 160.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL Corrective Action: Flag data outside of limit- Address in the project narrative	—	Not Applicable
Specific Conductance	Method Blank	120.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Specific Conductance (continued)	Laboratory Control Sample	120.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1	Not Applicable	9050A	Not Applicable
	Matrix Spike Duplicate	120.1	Not Applicable	9050A	Not Applicable
	Duplicate	120.1	Frequency: 1 with each batch of 20 samples processed Criteria: RPD[3] must be within laboratory QC limits Corrective Action: Flag associated data if outside of limits	9050	Frequency: 1 with each batch of 20 samples processed Criteria: RPD[3] must be within laboratory QC limits Corrective Action: Flag associated data if outside of limits
Sulfate	Method Blank	300.0[5] 375.1 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9038 9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Sulfate (continued)	Laboratory Control Sample	300.0[5] 375.1 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9038 9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Method 9038 Criteria: Percent recovery must be within ± 15 % Method 9056 Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)
	Matrix Spike	300.0[5] 375.1 375.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056	Frequency: 1 with each batch of samples processed not to exceed 10 samples (Method 9038) or 20 samples (Method 9056) Method 9038 Criteria: Limits are 75% - 125% recovery Method 9056 Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0[5] 375.1 375.4	Not Applicable	9038 9056	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Sulfate (continued)	Duplicate	300.0[5] 375.1 375.4	Not Applicable	9038 9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Sulfide	Method Blank	376.1 376.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9030B 9034	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	376.1 376.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9030B 9034	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Sulfide (continued)	Matrix Spike	376.1 376.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030B 9034	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	376.1 376.2	Not Applicable	9030B 9034	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 9034: Not Applicable
	Duplicate	376.1 376.2	Not Applicable	9030B 9034	Not Applicable
Sulfite	Method Blank	377.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Sulfite (continued)	Laboratory Control Sample	377.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	377.1	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	377.1	Not Applicable	—	Not Applicable
	Duplicate	377.1	Not Applicable	—	Not Applicable
Temperature	Method Blank	170.1	Not Applicable	---	Not Applicable
	Laboratory Control Sample	170.1	Not Applicable	---	Not Applicable
	Matrix Spike	170.1	Not Applicable	---	Not Applicable
	Matrix Spike Duplicate	170.1	Not Applicable	---	Not Applicable
	Duplicate	170.1	Not Applicable	---	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Total Organic Carbon (TOC)	Method Blank	415.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9060	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9060	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Total Organic Carbon (TOC) (continued)	Matrix Spike Duplicate	415.1	Not Applicable	9060	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1	Not Applicable	9060	Not Applicable
Total Organic Halides (TOX)	Method Blank	SM 5320B [5] 450.1 [5]	Frequency: 1 with each set of 8 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9020B	Frequency: Run in duplicate between each group of 8 analytical determinations Criteria: Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-WC-0001
	Laboratory Control Sample	SM 5320B [5] 450.1 [5]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)	9020B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within 90-110% Corrective Action: Rerun all samples associated with unacceptable LCS (ICV) SOP NO. CORP-WC-0001

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Total Organic Halides (TOX) (continued)	Matrix Spike	SM 5320B [5] 450.1 [5]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	Frequency: 1 per batch of 10 samples Criteria: Must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001
	Matrix Spike Duplicate	SM 5320B [5] 450.1 [5]	Not Applicable	9020B	Not Applicable
	Duplicate	SM 5320B [5] 450.1 [5]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] limit Corrective Action: Flag data outside of limit.	9020B	Frequency: All samples will be analyzed in duplicate Criteria: ≤ 20 % RPD[3] limit if both the sample and sample duplicate results are > 10 X MDL. Corrective Action: Flag data outside of limit. SOP NO. CORP-WC-0001
Turbidity	Method Blank	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Turbidity (continued)	Laboratory Control Sample	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	180.1	Not applicable	—	Not Applicable
	Matrix Spike Duplicate	180.1	Not Applicable	—	Not Applicable
	Duplicate	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit Not Applicable.	—	Not Applicable
Water Content	Method Blank	—	Not Applicable	—	Not Applicable
	Laboratory Control Sample	—	Not Applicable	—	Not Applicable
	Matrix Spike	—	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Water Content (continued)	Matrix Spike Duplicate	—	Not Applicable	—	Not Applicable
	Duplicate	—	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	—	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.
GFAA and Flame AA Metals, Mercury by CVAA	Method Blank	200 series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0003	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0003
	Laboratory Control Sample	200 series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0003	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0003

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
GFAA and Flame AA Metals, Mercury by CVAA (continued)	Matrix Spike	200 series	Frequency: with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 % Corrective Action: Flag data associated with unacceptable MS. (See SOP NO. CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 % Corrective Action: Flag data associated with unacceptable MS. (See SOP NO. CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)
	Matrix Spike Duplicate	200 series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 %, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable MSD SOP NO. CORP-MT-0003	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 %, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable MSD SOP NO. CORP-MT-0003
	Duplicate	200 series	Not Applicable	7000A series	Not Applicable
	Post Digestion Spikes	200 series	Post Digestion Spike is conducted on all samples	7000A series	Post Digestion Spike is conducted on all samples
ICP Metals	Method Blank	200.7	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0001	6010B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0001

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
ICP Metals (continued)	Laboratory Control Sample	200.7	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 85-115% Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0001	6010B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0001
	Matrix Spike	200.7	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001	6010B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001
	Matrix Spike Duplicate	200.7	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001	6010B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
ICP Metals (continued)	Duplicate	200.7	Not Applicable	6010B	Not Applicable
	Serial Dilution	200.7	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10 % Difference Corrective Action: Flag data associated with unacceptable Serial Dilution SOP NO. CORP-MT-0001	6010B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10 % Difference Corrective Action: Flag data associated with unacceptable Serial Dilution SOP NO. CORP-MT-0001
ICP/MS Metals	Method Blank	200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS	6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable blank
	Matrix Spike	200.8	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Recovery within laboratory control limits Corrective Action: Qualify data "suspect/matrix"	6020	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846) [2]
ICP/MS Metals (continued)	Matrix Spike Duplicate	200.8	Not Applicable	6020	Not Applicable
	Duplicate	200.8	Not Applicable	6020	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: 20% RPD[3] Corrective Action: Re-analyze samples associated with unacceptable duplicate
	Post Duplicate Spike	200.8	Not Applicable	6020	Frequency: 1 per 20 samples Criteria: 75-125% Corrective Action: Dilute and reanalyze
	Serial 5x Dilution	200.8	Not Applicable	6020	Frequency: 1 per 20 samples Criteria: ± 10% D Corrective Action: Use alternate isotope or quality data "suspect/matrix"

Footnotes

(1) National Pollutant Discharge Elimination System

(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/ChemicalMethods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

(3) RPD-Relative Percent Difference

(4) Orthophosphate only

(5) Method not listed in 40 CFR Part 136.

(6) Current promulgated method is a Guidance Method Only, SW-846, Final Update III, Rev.3, 12/96.

TABLE 8.4-6
Organic Laboratory Quality Control Samples

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Aromatic Volatiles by GC	Method Blank	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-GC-0001
	Laboratory Control Sample	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-GC-0001
	Matrix Spike	602	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-GC-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Aromatic Volatiles by GC (continued)	Matrix Spike Duplicate	602	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-GC-0001
	Duplicate	602	Not Applicable	8021B	Not Applicable
	Surrogates	602	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanlayze samples or flag sample data not meeting surrogate criteria SOP NO. CORP-GC-0001
	Internal Standards	602	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate. SOP NO. CORP-GC-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Acrolein &Acrylonitrile by GC	Method Blank	603	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8031 (Acrylo-nitrile only)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	603	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8031 (Acrylo-nitrile only)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	603	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8031 (Acrylo-nitrile only)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Acrolein & Acrylonitrile by GC (continued)	Matrix Spike Duplicate	603	Not Applicable	8031 (Acrylo-nitrile only)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	603	Not Applicable	8031 (Acrylo-nitrile only)	Not Applicable
	Surrogates	603	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8031 (Acrylo-nitrile only)	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	603	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8031 (Acrylo-nitrile only)	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Dioxins/ Dibenzo-furans (LRMS)	Method Blank	613	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Rerun all positive samples associated with unacceptable blank	8280A	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Rerun all positive samples associated with unacceptable blank
	Laboratory Control Sample	613	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8280A	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	613	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8280A	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	613	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable matrix spike Duplicate	8280A	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable matrix spike duplicate

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Dioxins/ Dibenzo-furans (LRMS) (continued)	Duplicate	613	Not Applicable	8280A	Not Applicable
	Surrogates	613	Not Applicable	8280A	Not Applicable
	Internal Standards	613	Internal standards are added to all samples (QC samples included). Internal standard recovery should be between 40 % to 120 %.	8280A	Internal standards are added to all samples (QC samples included). Internal standard recovery should be between 40 % - 120 % for Method 8280A. Use limits in laboratory SOP.
Dioxins/ Dibenzo-furans (HRGC/HR MS)	Method Blank	1613B[5]	Frequency: 1 per batch ≤ 20 samples extracted Criteria: Concentration less than reporting level or one third regulatory level whichever is greater Corrective Action: Rerun all positive samples associated with unacceptable blank	8290	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: Concentration less than reporting limit Corrective Action: Rerun all positive samples associated with unacceptable blank
	Laboratory Control Sample (Ongoing Precision and Recovery - OPR)	1613B[5]	Frequency: 1 per batch ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS (OPR)	8290	Frequency: 1 per batch of ≤ 20 samples extracted Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846) [2]
Dioxins/ Dibenzo-furans (HRGC/HR MS) (continued)	Matrix Spike	1613B[5]	Not Applicable	8290	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	1613B[5]	Not Applicable	8290	Frequency: 1 per analytical batch of ≤ 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable matrix spike duplicate
	Duplicate	1613B[5]	Not Applicable	8290	Not Applicable
	Surrogates	1613B[5]	Not Applicable	8290	Not Applicable
	Internal Standards (Labeled Compounds and Cleanup Standards)	1613B[5]	Labeled internal standards and cleanup standards are added to all samples (QC samples included). Recovery of each labeled standard should be within the method limits.	8290	Internal standards are added to all samples (QC samples included). Internal standard recovery should be between 40 % - 135 % for Method 8290. Use limits in laboratory SOP.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
PCBs (HRGC/HRMS)	Method Blank	1668[5]	Frequency: 1 per batch ≤ 20 samples extracted Criteria: Concentration less than reporting level or one third regulatory level whichever is greater Corrective Action: Rerun all positive samples associated with unacceptable blank	--	Not Applicable
	Laboratory Control Sample (Ongoing Precision and Recovery - OPR)	1668[5]	Frequency: 1 per batch ≤ 20 samples extracted Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS (OPR)	--	Not Applicable
	Matrix Spike	1668[5]	Not Applicable	--	Not Applicable
	Matrix Spike Duplicate	1668[5]	Not Applicable		Not Applicable
	Duplicate	1668[5]	Not Applicable	--	Not Applicable
	Surrogates	1668[5]	Not Applicable	--	Not Applicable

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846) [2]
PCBs (HRGC/HR MS) (continued)	Internal Standards (Labeled Compounds and Cleanup Standards)	1668[5]	Labeled internal standards and cleanup standards are added to all samples (QC samples included). Recovery of each labeled standard should be within the method limits.	–	Not Applicable
Halogenated Volatiles Volatiles by GC	Method Blank	–	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-GC-0001
	Laboratory Control Sample	–	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-GC-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Halogenated Volatiles by GC (continued)	Matrix Spike	--	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-GC-0001
	Matrix Spike Duplicate	--	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-GC-0001
	Duplicate	--	Not Applicable	8021B	Not Applicable
	Surrogates	--	Not Applicable	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria. SOP NO. CORP-GC-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Halogenated Volatiles by GC (continued)	Internal Standards	--	Not Applicable	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate. SOP NO. CORP-GC-0001
Herbicides	Method Blank	615[3]	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Re-extract all samples associated with unacceptable blank	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Re-extract all samples associated with unacceptable blank SOP NO. CORP-GC-0001
	Laboratory Control Sample	615[3]	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract all samples associated with unacceptable LCS	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS SOP NO. CORP-GC-0001
	Matrix Spike	615[3]	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-GC-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Herbicides (continued)	Matrix Spike Duplicate	615 [3]	Not Applicable	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike sample SOP NO. CORP-GC-0001
	Duplicate	615 [3]	Not Applicable	8151A	Not Applicable
	Surrogates	615 [3]	Not Applicable	8151A	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria SOP NO. CORP-GC-0001
	Internal Standards	615 [3]	Not Applicable	8151A	Optional
Nitro-aromatics by HPLC	Method Blank	--	Not Applicable	8330	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitro-aromatics by HPLC (continued)	Laboratory Control Sample	--	Not Applicable	8330	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	Not Applicable	8330	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	Not Applicable	8330	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	--	Not Applicable	8330	Not Applicable
	Surrogates	--	Not Applicable	8330	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	--	Not Applicable	8330	Not Applicable

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Organo-phosphorus Pesticides	Method Blank	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable MS
	Matrix Spike Duplicate	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable MS
	Duplicate	--	Not Applicable	8141A	Not Applicable

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Organo-phosphorus Pesticides (continued)	Surrogates	--	Not Applicable	8141A	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: Results must fall within laboratory-established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
PAHs by GC and HPLC	Method Blank	610	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8100 8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	610	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8100 8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
PAHs by GC and HPLC (continued)	Matrix Spike	610	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8100 8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	610	Not Applicable	8100 8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	610	Not Applicable	8100 8310	Not Applicable
	Surrogates	610	Not specified in method	8100 8310	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	610	Optional	8100 8310	Optional

TABLE 8.4-6
Organic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Pesticides/ PCBs	Method Blank	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reprepare and reanalyze all samples associated with unacceptable blank, see SOP CORP-GC-0001
	Laboratory Control Sample	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-GC-0001
	Matrix Spike	608	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike, see SOP CORP-GC-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Pesticides/ PCBs (continued)	Matrix Spike Duplicate	608	Not Applicable	8081A 80882	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike, see SOP CORP-GC-0001
	Duplicate	608	Not Applicable	8081A 8082	Not Applicable
	Surrogates	608	Not specified in method	8081A 8082	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria, see SOP CORP-GC-0001
	Internal Standards	608	Optional	8081A 8082	Optional

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846) [2]
Petroleum Hydro-carbons/Oil and Grease	Method Blank	413.1 413.2 418.1	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank Method 413.1: Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Petroleum Hydro-carbons/Oil and Grease (continued)	Laboratory Control Sample	413.1 413.2 418.1	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples Method 413.1: Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	413.1 413.2 418.1	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 413.1: Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	413.1 413.2 418.1	Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated Method 9071: Not Applicable

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Petroleum Hydro-carbons/Oil and Grease (continued)	Duplicate	413.1 413.2 418.1	Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated Method 9070: Not Applicable
	Surrogates	413.1 413.2 418.1	Not Applicable	9070 9071A	Not Applicable
	Internal Standards	413.1 413.2 418.1	Not Applicable	9070 9071A	Not Applicable
Petroleum Hydro-carbons	Method Blank	1664[4]	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-WC-0003	---	---

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Petroleum Hydro-carbons (continued)	Laboratory Control Sample	1664[4]	Frequency: 1 with each analytical batch Criteria: Waters - See limits in Table 2 of SOP No.: CORP-WC-0003 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-WC-0003	---	---
	Matrix Spike	1664[4]	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in Table 2 of SOP No. CORP-WC-0003 Corrective Action: See Section 9.6.1 of SOP No. CORP-WC-0003	---	---
	Matrix Spike Duplicate	1664[4]	Frequency: 1 with every 10 samples per site Criteria: See percent recovery and RPD limits in Table 2 of SOP No. CORP-WC-0003 Corrective Action: See Section 9.6.1 of SOP No. CORP-WC-0003	---	---
	Duplicate	1664[4]	Not Applicable	---	---

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Purgeable Halocarbons by GC	Method Blank	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-GC-0001
	Laboratory Control Sample	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS. see Section 9.7, SOP No. CORP-GC-0001
	Matrix Spike	601	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike. see Section 9.8. SOP No. CORP-GC-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Purgeable Halocarbons by GC (continued)	Matrix Spike Duplicate	601	Not Applicable	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike, see Section 9.7, SOP No. CORP-GC-0001
	Duplicate	601	Not Applicable	8021B	Not Applicable
	Surrogates	601	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria, see Section 9.4, SOP No. CORP-GC-0001
	Internal Standards	601	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Semivolatiles	Method Blank	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reextract and reanalyze all samples associated with unacceptable blank, see Section 9.3, SOP No. CORP-MS-0001
	Laboratory Control Sample	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Reextract and reanalyze all samples associated with unacceptable LCS, see Section 9.5.2, SOP No. CORP-MS-0001
	Matrix Spike	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike, see Section 9.6, SOP No. CORP-MS-0001

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Semivolatiles (continued)	Matrix Spike Duplicate	625	Not Applicable	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike, see Section 9.6, SOP No. CORP-MS-0001
	Duplicate	625	Not Applicable	8270C	Not Applicable
	Surrogates	625	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8270C	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria, see Section 9.7.2, SOP No. CORP-MS-0001
	Internal Standards	625	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8270C	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Volatiles by GC/MS	Method Blank	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank, see Section 9.4, SOP No. CORP-MS-0002
	Laboratory Control Sample	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS, see Section 9.5, SOP No. CORP-MS-0002
	Matrix Spike	624	Frequency: 1 per $\leq$ 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike, see Section 9.6, SOP No. CORP-MS-0002

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Volatiles by GC/MS (continued)	Matrix Spike Duplicate	624	Not Applicable	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike, see Section 9.6, SOP No. CORP-MS-0002
	Duplicate	624	Not Applicable	8260B	Not Applicable
	Surrogates	624	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8260B	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria, see Section 9.3, SOP No. CORP-MS-0002
	Internal Standards	624	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8260B	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Footnotes

(1) National Pollutant Discharge Elimination System

(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/ChemicalMethods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

(3) Method not listed in 40 CFR Part 136.

(4) Method 1664 is a proposed method only, and has not been promulgated by the EPA. These requirements are from Quanterra® SOP Number CORP-WC-0003, "HEM/SGT-HEM by Method 1664".

(5) Method 1613, Rev B, October 1994, EPA 821-B-94-005, "Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS". Method 1668, Draft, October 4, 1995, "Toxic PCBs by Isotope Dilution HRGC/HRMS".

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples

Analysis	QC Sample	Method	Requirement
Cyanide, Total	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL or less than 10x sample concentration Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG, whichever is more frequent Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix Spike, perform post distillation spike at 2 x CRDL or 2x sample concentration whichever is greater
	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable
	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all associated data associated if duplicate results outside control limits
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
ICAP (excludes mercury)	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL or less than 10x sample concentration Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% except silver and antimony Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix perform post digestion spike at 2xCRDL or 2x sample concentration whichever is greater
	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable
	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all data associated with duplicate results outside control limits

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
ICAP (excludes mercury) (continued)	Serial Dilution	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: <10% D when sample concentration > 50x IDL Corrective Action: Flag all data associated with results outside control limits
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable.
GFAA (excludes mercury)	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL or less than 10x sample concentration Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% except silver and antimony Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix
	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
GFAA (excludes mercury) (continued)	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all associated data associated if duplicate results outside control limits
	Analytical Spike	ILM03.0 ILMO4.0	Frequency: 1 with each sample except matrix spike Criteria: Evaluate per method requirements Corrective action: Perform per method requirements
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable.
Mercury (CVAA)	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Mercury (CVAA) (continued)	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable
	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all associated data associated if duplicate results outside control limits
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable.
PCDD, PCDF	Method Blank	DFLM01.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Chemical interference or electronic noise must be less than 5% of the appropriate internal standard ion A peak that meets identification criteria must be less than 2% of the signal of the appropriate internal standard ion Corrective Action: Reprepare all samples with positive results or those not meeting all identification criteria associated with unacceptable blank
	Laboratory Control Sample	DFLM01.1	Not Applicable
	Matrix Spike	DFLM01.1	Frequency: 1 for each matrix analyzed for each SDG Criteria: 50-150% Corrective Action: Verify all calculations and spiking; no further action required
	Matrix Spike Duplicate	DFLM01.1	Not Applicable

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
PCDD, PCDF (continued)	Duplicate	DFLM01.1	Frequency: 1 for each matrix analyzed for each SDG Criteria: RPD ≤ 50% Corrective Action: Verify all calculations and spiking; no further action required
	Surrogates	DFLM01.1	Not Applicable
	Internal Standards	DFLM01.1	Frequency: Internal standards are spiked into all samples and QC samples Criteria: 25 - 150% Corrective Action: Re-extract and reanalyze all samples with unacceptable surrogate recoveries
Pesticides/PCBs	Method Blank	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each extraction procedure within each SDG, whichever is most frequent or whenever samples are extracted Criteria: Concentration < CRQL Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	OLM03.1	Not Applicable.
	Matrix Spike	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each extraction procedure or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries outside of advisory limits

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Pesticides/PCBs (continued)	Matrix Spike Duplicate	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method RPD between MS/MSD should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits
	Duplicate	OLM03.1	Not Applicable
	Surrogates	OLM03.1	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each surrogate in samples should be within 30-150% Percent recovery for each surrogate in the method blank must be 30-150% Corrective Action: Flag unacceptable surrogate recoveries in samples Re-extract all samples associated with unacceptable surrogate recoveries in the method blank
	Internal Standards	OLM03.1	Not Applicable.
	Method Blank	OLM01.8	Frequency: 1 with each case of samples received (up to 20 samples and including matrix spikes and reanalyses) that are of similar matrix (water or soil) or similar concentration (soil), or 1 with each 14 calendar day period (7 calendar day for 14-day data turnaround contracts) during which samples in a case are received), or 1 whenever samples are extracted by the same procedure (continuous liquid - liquid extraction or sonication) Criteria: Concentration < CRQL Corrective Action: Re-extract and re-analyze all samples associated with unacceptable blank

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Pesticides/PCBs (continued)	Laboratory Control Sample	OLM01.8	Not Applicable
	Matrix Spike	OLM01.8	Frequency: 1 with every 20 samples of each matrix Criteria: Percent recovery should be within advisory limits given in method Corrective Action: Flag data associated with matrix spike recoveries outside of advisory limits
	Matrix Spike Duplicate	OLM01.8	Frequency: 1 with every 20 samples of each matrix Criteria: Percent recovery and RPD should be within advisory limits given in method Corrective Action: Flag data associated with percent recovery or RPD outside of advisory limits
	Duplicate	OLM01.8	Not Applicable
	Surrogates	OLM01.8	Frequency: Spiked onto all samples and QC samples Criteria: Advisory limits are 60% - 150% Corrective Action: Flag surrogate recoveries outside of advisory limits
	Internal Standards	OLM01.8	Not Applicable
Semivolatiles by GC/MS	Method Blank	OLM03.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than CRQL except phthalates which must be ≤ 5x CRQL Corrective Action: Re-extract and re-analyze all samples associated with unacceptable blank
	Laboratory Control Sample	OLM03.1	Not Applicable.

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Semivolatiles by GC/MS (continued)	Matrix Spike	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike outside of advisory limits
	Matrix Spike Duplicate	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method RPD between MS/MSD should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits
	Duplicate	OLM03.1	Not Applicable
	Surrogates	OLM03.1	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each surrogate must be within limits given in method (one base/neutral and/or one acid surrogate may be outside of limits but not below 10%) Corrective Action: Flag data associated with unacceptable recoveries or reanalyze all samples with unacceptable surrogate recoveries as required in method
	Internal Standards	OLM03.1	Frequency: Internal Standards are spiked onto all samples and QC samples Criteria: Internal Standard areas must be within -50% to + 100% from the last daily calibration check standard Corrective Action: Reanalyze all samples with unacceptable areas

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Semivolatiles by GC/MS (continued)	Method Blank	OLM01.8	Frequency: 1 with each case of samples received (up to 20 samples) and including matrix spikes and reanalyses) that are of similar matrix (water or soil) or similar concentration (soil), or 1 with each 14 calendar day period (7 calendar day for 14-day data turnaround contracts) during which samples in a case are received, or 1 whenever samples are extracted by the same procedure (continuous liquid-liquid extraction or sonication) Criteria: Concentration ≤ CRQL except phthalates which must be < 5 x CRQL Corrective Action: Re-extract and re-analyze all samples associated with unacceptable blank
	Laboratory Control Sample	OLM01.8	Not Applicable
	Matrix Spike	OLM01.8	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each 14 day calendar period (7 calendar day period for 14 day data turnaround contracts) during which field samples in a case were received, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries outside of advisory limits
	Matrix Spike Duplicate	OLM01.8	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each 14 day calendar period (7 calendar day period for 14 day data turnaround contracts) during which field samples in a case were received, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method. RPD between MS/MSD should be within advisory limits given in method. Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Semivolatiles by GC/MS (continued)	Duplicate	OLM01.8	Not Applicable
	Surrogates	OLM01.8	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each surrogate must be within limits given in method (one base/neutral and/or one acid surrogate may be outside of limits, but not below 10%) Corrective Action: Flag data associated with unacceptable recoveries or reanalyze all samples with unacceptable surrogate recoveries as required in method
	Internal Standards	OLM01.8	Frequency: Internal Standards are spiked onto all samples and QC samples Criteria: Internal Standard areas must be within -50% to + 100% from the last daily calibration check standard Corrective Action: Reanalyze all samples with unacceptable areas
Volatiles by GC/MS	Method Blank	OLM03.1	Frequency: 1 per 12 hours Criteria: Concentration less than CRQL except methylene chloride, acetone, 2-butanone must be ≤ 5x CRQL Corrective Action: Reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	OLM03.1	Not Applicable
	Matrix Spike	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike outside of advisory limits

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Volatiles by GC/MS (continued)	Matrix Spike Duplicate	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method RPD between MS/MSD should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits
	Duplicate	OLM03.1	Not Applicable
	Surrogates	OLM03.1	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each surrogate must be within limits given in method Corrective Action: Reanalyze all samples with unacceptable surrogate recoveries
	Internal Standards	OLM03.1	Frequency: Internal Standards are spiked onto all samples and QC samples Criteria: Internal Standard areas must be within -50% to + 100% from the last daily calibration check standard Corrective Action: Reanalyze all samples with unacceptable Internal Standard areas
	Storage Blank	OLM03.1	Frequency: 1 per SDG Criteria: Concentration less than CRQL except methylene chloride, acetone, 2-butanone must be ≤ 5x CRQL Corrective Action: Narrate with corrective action plan

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Volatiles by GC/MS (continued)	Method Blank	OLM01.8	Frequency: 1 per 12 hours Criteria: Concentration less than CRQL except methylene chloride, acetone, and 2-butanone must be ≤ 5x CRQL Corrective Action: Re-purge and re-analyze all associated samples
	Laboratory Control Sample	OLM01.8	Not Applicable
	Matrix Spike	OLM01.8	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each 14 day calendar period (7 calendar day period for 14 day data turnaround contracts) during which field samples in a case were received, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries outside of advisory limits
	Matrix Spike Duplicate	OLM01.8	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each 14 day calendar period (7 calendar day period for 14 day data turnaround contracts) during which field samples in a case were received, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method. RPD between MS/MSD should be within advisory limits given in method. Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits
	Duplicate	OLM01.8	Not Applicable
	Surrogates	OLM01.8	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Re-analyze all samples with unacceptable surrogate recoveries

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Volatiles by GC/MS (continued)	Internal Standards	OLM01.8	Frequency: Internal Standards are spiked onto all samples and QC samples Criteria: Internal Standard areas must be within -50% to + 100% from the last daily calibration check standard Corrective Action: Re-analyze all samples with unacceptable Internal Standard areas
	Storage Blank	OLM01.8	Not Applicable

Notes:

SDG = Sample Delivery Group

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1	250 mL plastic or glass, Cool, 4°C, 14 days	—	Not Applicable
	Solid[5]	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Alkalinity	Water	100 mL	310.1 2320B	250 mL plastic or glass, Cool, 4°C, 14 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Ammonia	Water	400 mL	350.1	500 mL plastic or glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Biochemical Oxygen Demand (BOD)	Water	200 mL	405.1	1000 mL plastic or glass, Cool, 4°C 48 hours	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Bromide	Water	100 mL	300.0[7] 320.1 ASTM D1246-88	250 mL plastic or glass, No preservative required, 28 days	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Chemical Oxygen Demand (COD)	Water	100 mL	410.1 410.2 410.4	250 mL glass or plastic, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	300.0[7] 325.1 325.2 325.3 4500 Cl E	250 mL plastic or glass, No preservative required, 28 days	9056 9251 9253	Method 9056: Cool, 4°C, analyze ASAP after collection. Method 9251/9253: 250ml plastic or glass, no preservative required, 28 days
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Chlorine, Residual	Water	100 mL	330.1 330.3	250 mL glass or plastic, Cool, 4°C, analyze immediately	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Chromium (Cr^{+6})	Water	100 mL	218.4 3500 Cr-D	Method 218.4: 200 mL plastic or glass, Cool, 4°C, 24 hours Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO_3 to pH <2 Cool, 4°C Analyze ASAP after collection	7196A	200 mL plastic or glass, Cool, 4°C, 24 hours
	Solid	Not Applicable	---	Not Applicable	3060A/ 7196A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Color	Water	100 mL	110.2	250 mL plastic or glass, Cool, 4°C, 48 hours	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Conductivity	Water	100 mL	120.1	200 mL glass or plastic, Cool, 4°C, 28 days	9050A	200 mL glass or plastic, Cool, 4°C, 24 hours
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Cyanide (Amenable)	Water	IL	335.1	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	9010B 9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days
	Solid	50g	—	Not Applicable	9010B 9012A	Not Specified
	Waste	50g	—	Not Applicable	9010B 9012A	Not Specified
Cyanide (Total)	Water	IL	335.1 335.2 335.3 4500 CN- D	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	9010B 9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C. 14 days
	Solid	50g	—	Not Applicable	9010B 9012A	8 or 16 oz glass Teflon-lined lids. Cool, 4°C, 14 days

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Cyanide (Total) (continued)	Waste	50g	--	Not Applicable	9010B 9012A	8 or 16 oz glass Teflon-lined lids, Cool, 4°C
Flashpoint (Ignitability)	Liquid	Not Applicable	---	Not Applicable	1010	No requirements, 250 mL amber glass, Cool, 4°C is recommended
	Solid	Not Applicable	--	Not Applicable	---	Not Applicable
	Waste	Not Applicable	--	Not Applicable	---	Not Applicable
Fluoride	Water	300 mL	300.0[7] 340.2	500 mL plastic, No preservation required, 28 days	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Iodide	Water	100 mL	345.1 Dionex	100 mL plastic or glass, Cool, 4°C, 24 hours		Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months This test should be performed in the field.	-	Not Applicable
	Solid	Not Applicable	-	Not Applicable	-	Not Applicable
	Waste	Not Applicable	-	Not Applicable	-	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Methylene Blue Active Substances (MBAS) (Surfactant)	Water	100 mL	425.1	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Nitrate	Water	100 mL	300.0[7] 352.1	Method 300.0: 250 mL plastic or glass, Cool, 4°C, 48 hours. Method 352.1: 250 mL plastic or glass, Cool, 4°C, 48 hours.	9056 9210	Method 9056: Cool, 4°C, analyze ASAP after collection Method 9210: Cool, 4°C Preserve by adding 1 mL of 1M boric acid solution per 100 mL of sample
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9210	Not Specified
Nitrite	Water	50 mL	300.0[7] 354.1	250 mL plastic or glass Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Nitrate-Nitrite	Water	100 mL	353.1 353.2 353.3	250 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Ortho-phosphate	Water	50 mL	300.0[7] 365.1 365.2 365.3 365.4	100 mL plastic or glass, Filter on site Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
pH	Water	50 mL	150.1 4500-H^+ B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**	9040B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**
	Solid	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.
	Waste	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic. Cool, 4°C. Analyze as soon as possible.
Phenolics	Water	100 mL	420.1 420.2	500 mL glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	9065 9066	1 liter glass recommended. Cool, 4°C. H_2SO_4 to pH < 4, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9065	Not Specified
Phosphate	Water	50 mL	---	Not Applicable	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	9056	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9056	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	50 mL	365.1 365.2 365.3 365.4	100 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Odor	Water	IL	140.1	200 mL glass only, Cool, 4°C, 24 hours		Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Reactivity (Cyanide and Sulfide)	Liquid	10 g	—	Not Applicable	Chapter 7 Sections 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
	Solid	10 g	—	Not Applicable	Chapter 7 Sections 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
	Waste	10 g	—	Not Applicable	Chapter 7 Sections 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass, Cool, 4°C, 48 hours	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Silica, Dissolved	Water	50 mL	370.1	Plastic only, 100 mL, Cool, 4°C, 28 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Specific Conductance	Water	50 mL	120.1	250 mL plastic or glass, Cool, 4°C, 24 hours	9050A	250 mL plastic or glass, Cool, 4°C, 28 days
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Sulfate (SO_4)	Water	100 mL	300.0[7] 375.1 375.4	100 mL plastic or glass, Cool, 4°C, 28 days	9056 9038	Method 9056: Cool, 4°C, analyze ASAP collection Method 9038: 200 mL plastic or glass, Cool, 4°C, 28 days
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	100 mL	—	Not Applicable	9038	200 mL plastic or glass, Cool, 4°C, 28 days
Sulfide	Water	100 mL	376.1 376.2	500 mL plastic or glass, Cool, 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030B 9034	500 mL plastic, no headspace, Cool, 4°C, Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	—	Not Applicable	9030B 9034	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free
	Waste	50 g	—	Not Applicable	9030B 9034	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Sulfite (SO_3)	Water	100 mL	377.1	100 mL plastic or glass, No preservative required, analyze immediately **This test should be performed in the field.**	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Temperature	Water	200 mL	170.1	1 liter plastic or glass, analyze immediately in the field	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Total Dissolved Solids (Filterable)	Water	100 mL	160.1	250 mL plastic or glass, Cool, 4°C, 7 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Total Kjeldahl Nitrogen (TKN)	Water	500 mL	351.2 351.3	500 mL plastic or glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Total Organic Carbon (TOC)	Water	100 mL	415.1	100 mL plastic or glass. Cool, 4°C, H_2SO_4 to pH < 2, 28 days	9060	100 mL glass or 40 mL VOA vials. Cool, 4°C. H_2SO_4 or HCl to pH < 2. 28 days
	Solid	Not Applicable	—	Not Applicable	9060	Not Specified
	Waste	Not Applicable	—	Not Applicable	9060	Not Specified

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Total Organic Halides (TOX)	Water	100 mL	5320B [7] 450.1 [7]	Method 5320B: 500 mL amber glass, Teflon®-lined lid, Cool, 4°C, HNO_3 to pH <2, no headspace, 14 days Method 450.1: 500 mL amber glass, Teflon®-lined lid, Cool, 4°C, HNO_3 to pH <2, no headspace, 28 days	9020B	500 mL amber glass, Teflon®-lined lid, Cool, 4°C, H_2SO_4 to pH < 2, no headspace, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Solids	Water	100 mL	160.3	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Turbidity	Water	50 mL	180.1	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Water Content	Water	Not Applicable	—	Not Applicable	—	Not Applicable
	Solid	10 g	—	Refer to specific method used	—	Refer to specific method used
	Waste	10 g	—	Refer to specific method used	—	Refer to specific method used
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months	6010B, 6020, 7000A series	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months
	Solid	200 g	200 series	8 or 16 oz glass or polyethylene container storage at 4 °C	6010B, 6020, 7000A series	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	Not Applicable	6010B, 6020, 7000A series	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA)	Water	100 mL	245.1	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days
	Solid	200 g	245.5	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	Not Applicable	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
(2) National Pollutant Discharge Elimination System - MCAWW, March 1983.
(3) Holding times are calculated from date of collection.
(4) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(5) Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.
(6) Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.
(7) Method not listed in 40 CFR Part 136.

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH < 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2
	Solid[5]	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C (Preservation and holding times are subject to client specifications)[12]
	Waste	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis.

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	Method	NPDES[2], [3]		RCRA (SW846)[3], [4]
				Requirements	Method[6]	Requirements
Aromatic Volatiles (continued)	Waste	5 g or 25 g	--	Not Applicable	8021B	Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C (Preservation and holding times are subject to client specifications) [12]
Acrolein & Acrylonitrile by GC	Water	40 mL	603	Glass with Teflon®-lined septum, Cool, 4 °C (± 2°C), 0.008% $Na_2S_2O_3$[11], Adjust pH 4-5[8] 14 days	8031 (Acrylo-nitrile only)	Glass with Teflon®-lined septum, Cool, 4 °C (± 2°C), 0.008% $Na_2S_2O_3$[11], Adjust pH 4-5[8] 14 days
	Solid	-	-	Not Applicble	-	Not Applicable
	Waste	-	-	Not Applicble	-	Not Applicable
Dioxins/ Dibenzo-furans (LRMS)	Water	1L	613	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction, 7 days Analysis, 40 days	8280A	1 liter glass amber with Teflon®-lined lid, Cool 4°C, if residual chlorine is present in aqueous samples, add 80 mg of sodium thiosulfate per liter of sample, if sample pH >9, adjust to pH 7-9 with H_2SO_4, Extract within 30 days Analyze within 45 days of extraction
	Solid	10 g	--	Not Applicable	8280A	8 or 16 oz glass amber wide mouth with Teflon®-lined lid, Cool 4°C, Extract within 30 days Analyze within 45 days of extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Dioxins/ Dibenzo-furans (LRMS) (continued)	Waste	10 g	--	Not Applicable	8280A	8 or 16 oz glass amber wide mouth with Teflon®-lined lid, Cool 4°C, Extract within 30 days and Analyze within 45 days of extraction
	Water	1L	613	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction, 7 days Analysis, 40 days	--	Not Applicable
	Solid	10 g	--	Not Applicable	--	Not Applicable
	Waste	10 g	--	Not Applicable	--	Not Applicable
Dioxins/Dibenzo -furans (HRGC/HRMS)	Water	I L	1613B	1.1 liter amber glass w/fluoropolymer-lined screw cap. Add sodium thiosulfate if residual chlorine. If pH > 9, H_2SO_4 to pH 7-9. Cool, 0–4°C in the dark. Extraction , 1 year. Analysis, 1 year.	8290	1 liter glass amber with Teflon®-lined lid, Cool, 4°C, Extraction, 30 days Analysis, 45 days from date of extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Dioxins/Dibenzo -furans (HRGC/HRMS) (continued)	Solid	10g	1613B	500 mL amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, 1 year.	8290	8 or 16 oz glass amber wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 30 days Analysis, 45 days from date of extraction
	Waste	10g	1613B	500 mL amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, 1 year.	8290	8 or 16 oz glass amber wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 30 days Analysis, 45 days from date of extraction
PCBS By HRGC/HRMS	Water	1 L	1668	1.1 liter amber glass w/fluoropolymer-lined screw cap. Add sodium thiosulfate if residual chlorine. H_2SO_4 to pH 2-3. Cool, 0-4°C in the dark. Extraction , 1 year. Analysis, 1 year.	--	Not Applicable
	Solid	10g	1668	500 mL amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, 1 year.	--	Not Applicable

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
PCBS By HRGC/HRMS (continued)	Waste	10g	1668	500 mL amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, 1 year.	–	Not Applicable
Halogenated Volatiles By GC	Water	40 mL	–	Not Applicable	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days
	Solid[5]	5 g or 25 g	–		8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C (Preservation and holding times are subject to client specifications) [12]

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Halogenated Volatiles (continued)	Waste	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C (Preservation and holding times are subject to client specifications) [12]
Herbicides	Water	1L	615 [10]	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present, Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	--	Not Applicable	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Herbicides (continued)	Waste	50 g	--	Not Applicable	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool 4 °C Extraction, 14 days Analysis, 40 days of the start of the extraction
Nitroaromatics	Water	1L	--	Not Applicable	8330	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	—	Not Applicable	8330	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Nitroaromatics (continued)	Waste	50 g	—	Not Applicable	8330	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction
Organo-phosphorus Pesticides	Water	1L	—	Not Applicable	8141A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	—	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	—	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
PAHs by GC and HPLC	Water	1L	610	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8100 8310	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
PAHs by GC and HPLC (continued)	Solid	50 g	—	Not Applicable	8100 8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	—	Not Applicable	8100 8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	—	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	—	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Petroleum Hydrocarbons/ Oil and Grease	Water	1L	413.1 413.2 418.1	1 liter glass, Cool, 4°C, HCl to pH <2, 28 days	9070	1 liter glass with Cool, 4°C, HCl to pH <2, 28 days
	Solid	---	---	Not Applicable	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
	Waste	---	---	Not Applicable	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
	Water	1 L	1664[7]	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days	---	---
	Solid	30 g	1664[7]	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days	---	---
	Waste	---	---	Not Applicable	---	---
Purgeable Halocarbons By GC	Water	40 mL	601	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine present, 14 days	8021B	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 1:1 HCl to pH ≤ 2, sodium thiosulfate if residual chlorine present, 14 days
	Solid	5 g or 25 g	---	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis.

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Purgeable Halocarbons By GC (continued)	Solid	5 g or 25 g	—	Not Applicable	8021B	Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C (Preservation and holding times are subject to client specifications) [12]
	Waste	5 g or 25 g	—	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate).Cool, 4°C (Preservation and holding times are subject to client specifications) [12]

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction, 7 days Analysis, 40 days	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, within 40 days of extraction
	Solid	50 g	—	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
	Waste	50 g	—	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2[8]	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[9]

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Volatile Organics (continued)	Solid[5]	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C (Preservation and holding times are subject to client specifications)
	Waste	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C (Preservation and holding times are subject to client specifications)

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
(2) National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.
(3) Holding times are calculated from the date of collection.
(4) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(5) Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.
(6) Only one determination method is listed when separate methods are required for preparation and analysis.
(7) **Method 1664 is a proposed only method and has not been promulgated by the EPA.**
(8) **For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.**
(9) **For acrolein and acrylonitrile the pH should be adjusted to 4-5.**
(10) Method not listed in 40 CFR Part 136.
(11) Should only be used in the presence of residual chlorine.
(12) Data from Encore shows that with freezing, the sample in an Encore may be held for up to 7 days. Until formal EPA approval, this holding time must be approved by the client.

TABLE 8.5-3
Radiological Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Recommended Containers[1]	Preservative	Maximum Holding Time	Minimum Volume Required for Analysis[2]
Gross Alpha/Beta	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	500 mls
	Soil	P, G	None		50[4] gms
Americium-241	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Carbon-14	Water	P, G	Field adjusted to pH > 9 with NaOH[3]	180 days after collection	100 mls
	Soil	P, G	None		50[4] gms
Calcium-45	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	100 mls
Curium-242	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Gamma Emitters Actinides, as applicable, Co-60, Cs-137, K-40, Mn-54, and other fission/activation products	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		650[7] gms
Iron-55	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	50 mls
Lead-210	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	500 mls
	Soil	P, G	None		50[4] gms
Neptunium-237	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Promethium-147	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	250 mls

TABLE 8.5-3
Radiological Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Recommended Containers[1]	Preservative	Maximum Holding Time	Minimum Volume Required for Analysis[2]
Plutonium-238, 239/240	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Radium-226	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Radium-228	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Strontium-89, 90 and Total Strontium	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Technetium-99	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	100 mls
	Soil	P, G	None		50[4] gms
Thorium-227, 228, 230, 232	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Total Uranium	Water	P,G	Field acidified to pH < 2 with HNO_3	180 days after collection	50 mls
	Soil	P, G	None		50[4] gms
Tritium	Water	P, G[6]	None	180 days after collection	100 mls
	Soil	P, G[6]	None		100 gms
Uranium-233/234, 235/236	Water	P, G	Field acidified to pH < 2 with HNO_3	180 days after collection	1000[5] mls
	Soil	P, G	None		50[4] gms
Uranium-238	Soil	P, G	None	180 days after collection	50[4] gms

TABLE 8.5-3
Radiological Sample Containers, Preservatives, and Holding Times (Continued)

Footnotes

(1) Plastic (polyethylene), Glass
(2) Assumes that quality control samples have been assigned in the field. If duplicates, matrix spikes and/or matrix spike duplicates are to be assigned by the laboratory, additional multiple sample volumes are required. Volumes listed are for standard aliquot size. Detection limit requirements may necessitate larger volumes.
(3) Assumes that carbon is in the form of CO_3^{--}.
(4) May be aliquoted or sequentially determined from the same volume.
(5) May be aliquoted or sequentially determined from the same volume.
(6) Tritium is very volatile. Sample containers must be air tight to eliminate tritium loss.
(7) Dry weight.

TABLE 8.5-4
Sample Containers, Preservatives, and Holding Times for USEPA Contract Laboratory Program Statement of Work

Analytical Parameters	Matrix	Minimum Sample Size	Requirements[1]
Cyanide, Total and Amenable to Chlorination	Water	500 mL	500 mL, glass or polyethylene container, 0.6 g ascorbic acid (only in presence of residual chlorine) NaOH to pH > 12, Cool, 4°C, 12 days
	Soil/Sediment	25 g	8 or 16 oz glass with Teflon-lined lids, Cool, 4°C, 12 days
ICAP and GFAA (excludes mercury)	Water	100 mL	1 liter glass or polyethylene container, HNO_3 to pH =2, 180 days
	Soil/Sediment	25 g	4 or 8 oz glass or polyethylene container, Cool, 4°C, 180 days
Mercury (CVAA)	Water	100 mL	1 liter glass or polyethylene container, HNO_3 to pH =2, 26 days
	Soil/Sediment	25 g	8 or 16 oz glass with Teflon®-lined lids, Cool, 4°C, 26 days
Pesticides/PCBs	Water	1 L	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction within 5 days of sample receipt Analysis within 40 days after start of extraction
	Soil/Sediment	50 g	8 or 16 oz glass wide mouth with Teflon®-lined lid, protect from light, Cool, 4°C, Extraction within 10 days of sample receipt Analysis within 40 days after start of extraction
PCDD, PCDF[2]	Water	1 L	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, No holding time requirements specified
	Soil/Sediment/ Fly Ash/ Chemical Waste	25 g	4 or 8 oz glass wide mouth with Teflon®-lined lid, protect from light, room temperature, No holding time requirements specified

TABLE 8.5-4
Sample Containers, Preservatives, and Holding Times for USEPA Contract Laboratory Program Statement of Work (Continued)

Analytical Parameters	Matrix	Minimum Sample Size	Requirements[1]
Semivolatiles	Water	1L	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction within 5 days of sample receipt Analysis within 40 days after start of extraction
	Soil/Sediment	50 g	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction within 10 days of sample receipt Analysis within 40 days after start of extraction
Volatiles	Water	40 mL	40 mL glass with Teflon®-lined lid, no entrapped air bubbles pH <2[3], Cool, 4°C, 10 days
	Soil/Sediment	25 g	4 or 8 oz glass with Teflon®-lined lids, Cool, 4°C, 10 days

Footnotes

(1) Holding times are calculated from verified time of sample receipt.

(2) PCDD: Polychlorinated Dibenzo-p-dioxins
PCDF: Polychlorinated Dibenzofurans

(3) The OLM03.0 requirement is to acidify the sample to pH<2. The OLM01.8 requirement is to determine and report the pH of the sample to check that the sample was acidified in the field.

TABLE 8.5-5
Sample Containers, Preservatives, and Holding Times for TCLP[1] and SPLP[2]

Analytical Parameters	Matrix	Minimum Sample Size[3]	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

(1) TCLP = Toxicity Characteristic Leaching Procedure

(2) SPLP = Synthetic Precipitation Leaching Procedure

(3) Smaller sample size is adequate for solid samples or individual fractions. A combined volume of 32 oz. is recommended for semivolatiles and metals. A separate 4 oz. container should always be used for the volatile fraction. Volatile fractions should be stored with minimal headspace.

TABLE 8.5-6
Periodic Equipment Calibrations

Type of Equipment	Calibration Requirements
Balances	• Must be serviced and calibrated annually by an approved vendor • Calibration must be checked daily or before use by analyst with weight(s) classified as Class "S" (or Class "S" traceable) by NIST per operation-specific SOPs. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in log. • All Class "S" weights must be certified by an outside vendor every three years.
Thermometers	• Working glass thermometers must be calibrated against a certified NIST thermometer at least annually as described in operation-specific SOPs. • Working non-glass thermometers must be calibrated against a certified NIST thermometer quarterly as described in operation-specific SOPs. • The NIST thermometer must be recertified every three years.
Refrigerators/Freezers	• Thermometers must be immersed in a liquid such as mineral oil or glycol • Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: 4°C ± 2°C Freezer acceptance limits: < - 10°C
Ovens	• Temperature of units must be checked daily or before use. • Acceptance limits vary according to use as described in operation-specific SOPs and must be documented in the temperature log.
Micropipettors	• Calibrations are checked gravimetrically as required by the operation-specific SOP. • Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.
Syringes, Volumetric Glassware and Graduated Glassware	• All syringes and volumetric glassware are purchased as Class A items. • Class A items are certified by the manufacturer to be within ± 1% of the measured volume, therefore, calibration of these items by Quanterra® laboratories is not required. • All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents and sample volumes are within method tolerances.

TABLE 8.5-7
Summary of Inorganic Method Calibrations

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Acidity	Initial	305.1	2 point calibration of pH meter (± 0.05 pH units of true value)	--	Not Applicable
	Continuing	305.1	Not Applicable	--	Not Applicable
	Ending	305.1	Not Applicable	--	Not Applicable
Alkalinity	Initial	310.1 2320B	2 point calibration of pH meter (± 0.05 pH units of true value)	--	Not Applicable
	Continuing	310.1 2320B	Not Applicable	--	Not Applicable
	Ending	310.1 2320B	Not Applicable	--	Not Applicable
Ammonia	Initial	350.1	6 levels including blank, "r"[3] ≥ 0.995	--	Not Applicable
	Continuing	350.1	1 level or LCS every 10 samples ± 10% of true value	--	Not Applicable
	Ending	350.1	1 level or LCS every 10 samples ± 10% of true value	--	Not Applicable
Biochemical Oxygen Demand (BOD)	Initial	405.1	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	Not Applicable
	Continuing	405.1	Not Applicable	--	Not Applicable
	Ending	405.1	Not Applicable	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Bromide	Initial	300.0[4] 320.1 ASTM D1246-88	Method 300.0 & ASTM D1246-88: 5 levels plus a blank, "r"[3] ≥ 0.995 Method 320.1: Not Applicable	9056	Method 300.0: 5 levels plus a blank, "r"[3] ≥ 0.995
	Continuing	300.0[4] 320.1 ASTM D1246-88	Method 300.0 & ASTM D1246-88:1 level every 10 samples ± 10% of true value Method 320.1 Not Applicable	9056	Method 300.0: Not Applicable
	Ending	300.0[4] 320.1 ASTM D1246-88	Not Applicable	9056	Not Applicable
Chemical Oxygen Demand (COD)	Initial	410.4 410.1 410.2	Method 410.4: 5 levels plus a blank"r"[3] ≥ 0.995 Methods 410.1 & 410.2: Standardize titrant.	--	Not Applicable
	Continuing	410.4 410.1 410.2	Method 410.4: 1 level every 10 samples ± 10% of true value Methods 410.1 & 410.2: Not Applicable	--	Not Applicable
	Ending	410.4	Method 410.4: 1 level ± 10% of true value Methods 410.1 & 410.2: Not Applicable	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Chloride	Initial	300.0[4] 325.1 325.2 325.3 4500-Cl E	Method 300.0, 325.1, and 325.2, 4500 Cl E: 5 levels plus blank "r"[3] ≥ 0.995 Method 325.3: Standardize titrant	9056 9251 9253	Method 9056: 3 levels plus a blank Method 9251: 5 levels plus blank "r"[3] ≥ 0.995 Method 9253: Standardize titrant
	Continuing	300.0[4] 325.1 325.2 325.3 4500-Cl E	Method 300.0, 325.1, and 325.2, 4500-Cl E: 1 level every 10 samples ± 10% of true value Method 325.3: Not Applicable	9056 9251 9253	Method 9056: 1 per batch of 20 samples, ± 10% of true value Method 9251: 1 level every 10 samples ± 10% of true value Method 9253: Not Applicable
	Ending	300.0[4] 325.1 325.2 325.3 4500-Cl E	Method 300.0, 325.1, and 325.2, 4500-Cl E: 1 level every 10 samples ± 10% of true value Method 325.3: Not Applicable	9056 9251 9253	Method 9056 and 9253: Not Applicable Method 9251: 1 level ± 10% of true value
Chromium Cr^{+6}	Initial	218.4 3500 Cr-D	Method 218.4: 5 levels plus blank "r"[3] ≥ 0.995 Method 3500 Cr-D: 3 levels plus blank	7196A	5 levels plus blank "r"[3] ≥ 0.995
	Continuing	218.4 3500 Cr-D	1 level every 10 samples ± 10% of true value	7196A	1 level every 10 samples ± 15%
	Ending	218.4 3500 Cr-D	1 level ± 10% of true value	7196A	1 level ± 15%

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Chlorine, Residual	Initial	330.1 330.3	Standardize titrant	--	Not Applicable
	Continuing	330.1 330.3	Not Applicable	--	Not Applicable
	Ending	330.1 330.3	Not Applicable	--	Not Applicable
Color	Initial	110.2	3 levels plus blank	--	Not Applicable
	Continuing	110.2	1 level every 10 samples	--	Not Applicable
	Ending	110.2	1 level	--	Not Applicable
Conductivity	Initial	120.1	Standard KCl solution	9050A	1 level to determine cell constant
	Continuing	120.1	Not Applicable	9050A	Not Applicable
	Ending	120.1	Not Applicable	9050A	Not Applicable
Cyanide (Amenable)	Initial	335.1	7 levels plus blank "r"[3] ≥ 0.995	9010B 9012A	7 levels plus blank "r"[3] ≥ 0.995
	Continuing	335.1	1 level every 10 samples ± 10% of true	9010B 9012A	1 mid-level every 10 samples ± 15% of true value
	Ending	335.1	1 level ± 10 % of true value	9010B 9012A	± 15% of true value
Cyanide (Total)	Initial	335.1 335.2 335.3 4500-CN E	7 levels plus blank "r"[3] ≥ 0.995	9010B 9012A	7 levels plus blank "r"[3] ≥ 0.995
	Continuing	335.1 335.2 335.3 4500-CN E	1 mid-level every 10 samples ± 10 % of true value	9010B 9012A	1 mid-level every 10 samples ± 15% of true value
	Ending	335.1 335.2 335.3 4500-CN E	1 mid-level ± 10 % of true value	9010B 9012A	± 15% of true value

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Flashpoint	Initial	--	Not Applicable	1010	p-Xylene reference standard must have flashpoint of 27.2°C ± 1.1°C
	Continuing	--	Not Applicable	1010	Not Applicable
	Ending	--	Not Applicable	1010	Not Applicable
Fluoride	Initial	300.0[4] 340.2	Method 300.0: 5 levels plus a blank, "r"[3] ≥ 0.995 Method 340.2: 6 levels "r"[3] ≥ 0.995	9056	3 levels plus a blank
	Continuing	300.0[4] 340.2	1 mid-level every 10 samples ± 10% of true value	9056	1 per batch of 20 samples ± 10% of true value
	Ending	300.0[4] 340.2	1 mid-level ± 10% of true value	9056	Not Applicable
Hardness	Initial	130.2 2340B	Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7	--	Not Applicable
	Continuing	130.2 2340B	Method 130.2: Not Applicable Method 2340B: See ICP Metals 200.7	--	Not Applicable
	Ending	130.2 2340B	Method 130.2: Not Applicable Method 2340B: See ICP Metals 200.7	--	Not Applicable
Iodide	Initial	345.1 Dionex[3]	Method 345.1: Standardize titrant Dionex: 4 levels plus blank "r"[3] ≥ 0.995	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Iodide (continued)	Continuing	345.1 Dionex[3]	Method 345.1: Not Applicable Dionex: 1 mid-level every 10 samples ± 10 % of true value	--	Not Applicable
	Ending	345.1 Dionex[3]	Method 345.1: Not Applicable Dionex: 1 mid-level ± 10% of true value	--	Not Applicable
Iron (Ferrous)	Initial	3500-Fe D	3 levels plus a blank, "r"[3] ≥ 0.995	-	Not Applicable
	Continuing	3500-Fe D	1 mid-level every 10 samples ± 10% of true value	-	Not Applicable
	Ending	3500-Fe D	1 mid-level ± 10% of true value	-	Not Applicable
Methylene Blue Active Substances (MBAS)	Initial	425.1	4 levels plus blank "r"[3] ≥ 0.995	--	Not Applicable
	Continuing	425.1	1 level every 10 samples ± 10 % of true value	--	Not Applicable
	Ending	425.1	1 level ± 10 % of true value	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Nitrate	Initial	300.0[4] 352.1	5 levels plus a blank "r"[3] ≥ 0.995	9056 9210	Method 9056: 3 levels plus a blank Method 9210: 2 levels plus a blank must be analyzed with each batch of 20 samples (ICV ± 10%)
	Continuing	300.0[4] 352.1	1 mid-level every 10 samples ± 10% of true value	9056 — 9210	Method 9056: 1 per batch of 20 samples, ± 10% of true value Method 9210: Independently - prepared check standard every 10 samples
	Ending	300.0[4] 352.1	1 mid-level ± 10% of true value	9056 — 9210	Method 9056: Not Applicable Method 9210: After final sample CCV must be analyzed
Nitrite	Initial	300.0[4] 354.1	5 levels plus a blank "r"[3] ≥ 0.995	9056	3 levels plus a blank
	Continuing	300.0[4] 354.1	1 mid-level every 10 samples ± 10% of true value	9056	1 per batch of 20 samples. ± 10% of true value
	Ending	300.0[4] 354.1	1 mid-level ± 10% of true value	9056	Not Applicable
Nitrate-Nitrite	Initial	353.1 353.2 353.3	5 levels plus blank "r"[3] ≥ 0.995	--	Not Applicable
	Continuing	353.1 353.2 353.3	1 level every 10 samples ± 10% of true value	--	Not Applicable
	Ending	353.1 353.2 353.3	1 mid-level ± 10% of true value	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Odor	Initial	140.1	No calibration	--	Not Applicable
	Continuing	140.1	Not Applicable	--	Not Applicable
	Ending	140.1	Not Applicable	--	Not Applicable
Phosphorus (total and Ortho-phosphate)	Initial	300.0[4] 365.1 365.2 365.3 365.4	Method 300.0/365.3/365.4: 3 levels plus a blank Method 365.2: 8 levels plus a blank	--	Not Applicable
	Continuing	300.0[4] 365.1 365.2 365.3 365.4	Method 300.0/365.3/365.4: 1 level every 10 samples ± 10% of true value Method 365.2: Blank and 2 standards with each series of samples, ± 2% of true value or recalibrate	--	Not Applicable
	Ending	300.0[4] 365.1 365.2 365.3 365.4	Method 300.0/365.3/365.4: ± 10% of true value Method 365.2: Not Applicable	--	Not Applicable
pH	Initial	150.1 4500-H^+ B	2 level calibration that bracket the expected pH of the sample (± 0.05 pH units of true value)	9040B 9045C	2 point calibration (± 0.05 pH units of true value)
	Continuing	150.1 4500-H^+ B	1 buffer check every 10 samples ± 5% of true value	9040B 9045C	Not Applicable
	Other	150.1 4500-H^+ B	Third point check	9040B 9045C	Third point check

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
pH (continued)	Ending	150.1 4500-H^+ B	1 buffer check ± 5% of true value	9040B 9045C	Not Applicable
Phenolics	Initial	420.1 420.2	5 levels plus a blank "r"[3] ≥ 0.995	9065 9066	5 levels plus a blank "r"[3] 0.995
	Continuing	420.1 420.2	1 mid-level every 10 samples ± 10% true value	9065 9066	1 mid-level ± 15% true value
	Ending	420.1 420.2	1 mid-level ± 10% true value	9065 9066	1 mid-level ± 15% true value
Phosphate	Initial	---	Not Applicable	9056	3 levels plus a blank
	Continuing	---	Not Applicable	9056	1 per batch of 20 samples, ± 15% of true value
	Ending	---	Not Applicable	9056	Not Applicable
Reactivity	Initial	--	Not Applicable	Chap 7	See Total Cyanide and Sulfide
	Continuing	--	Not Applicable		
	Ending	--	Not Applicable		
Settleable Solids	Initial	160.5	Not Applicable	--	Not Applicable
	Continuing	160.5		--	Not Applicable
	Ending	160.5		--	Not Applicable
Silica, Dissolved	Initial	370.1	Approximately 6 levels plus blank "r"[3] ≥ 0.995	--	Not Applicable
	Continuing	370.1	1 level ± 15% of true value	--	Not Applicable
	Ending	370.1	1 level ± 15% of true value	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Specific Conductance	Initial	120.1	Standardize meter with 0.01 M KCl	9050A	Not Applicable
	Continuing	120.1	1 level every 10 samples ± 10% of true value	9050A	Not Applicable
	Ending	120.1	1 level ± 10% of true value	9050A	Not Applicable
Sulfate	Initial	300.0[4] 375.1 375.4	Method 300.0/375.1: 5 levels plus blank "r"[3] ≥ 0.995 Method 375.4: 3 levels plus blank "r"[3] ≥ 0.995	9038 9056	Method 9038: 3 levels plus a blank for every hour of continuous sample analysis. Method 9056: 3 levels plus a blank
	Continuing	300.0[4] 375.1 375.4	Method 300.0/375.1: 1 mid-level after every 10 samples ± 10% of true value Method 375.4: 1 level every 3 or 4 samples ± 10% of true value	9038 9056	Method 9038: Independent-prepared check standard every 15 samples Method 9056: 1 per batch of 20 samples, ± 10% of true value
	Ending	300.0[4] 375.1 375.4	± 10% of true value	9038 9056	Not Applicable
Sulfide	Initial	376.1 376.2	Method 376.1: This is a titration method. Therefore, calibrations are not applicable. Method 376.2: 5 levels plus a blank "r" [3] ≥ 0.995	9030B 9034	This is a colorimetric titration. Therefore, calibration is not applicable.

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Sulfide (continued)	Continuing	376.1 376.2	Method 376.1: Not Applicable Method 376.2: 1 level every 10 samples ± 10% of true value	9030B 9034	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1 376.2	Method 376.1: Not Applicable Method 376.2: ± 10% of true value	9030B 9034	This is a colorimetric titration. Therefore, calibration is not applicable.
Sulfite	Initial	377.1	This is a colorimetric titration. Therefore, calibration is not applicable.	--	Not Applicable
	Continuing	377.1		--	Not Applicable
	Ending	377.1		--	Not Applicable
Temperature	Initial	170.1	Not Applicable	--	Not Applicable
	Continuing	170.1	Not Applicable	--	Not Applicable
	Ending	170.1	Not Applicable	--	Not Applicable
Total Dissolved Solids	Initial	160.1	This is a gravimetric determination. Calibrate balance prior to analysis	--	Not Applicable
	Continuing	160.1		--	Not Applicable
	Ending	160.1		--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.2 351.3	Method 351.2: 5 levels plus blank "r"[3] ≥ 0.995 Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	–	Not Applicable
	Continuing	351.2 351.3	Method 351.2: 1 mid-level every 10 samples ± 10% of true value Method 351.3: Not Applicable	–	Not Applicable
	Ending	351.2 351.3	Method 351.2: ± 10% of true value Method 351.3: Not Applicable	–	Not Applicable
Total Organic Carbon (TOC)	Initial	415.1	3 levels plus blank	9060	3 levels plus blank "r"[3] ≥ 0.995
	Continuing	415.1	1 mid-level every 10 samples ± 15% of true value	9060	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1	± 15% of true value	9060	± 15% of true value

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Total Organic Halides (TOX)	Initial	SM 5320B [4] 450.1 [4]	Method 5320B: 7 levels plus a blank ± 10% of true value Method 450.1: Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration std.) Verify with independently-prepared check standard	9020B	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration std.) Verify with independently-prepared check standard – ICV ± 10% SOP NO. CORP-WC-0001
	Continuing	SM 5320B [4] 450.1 [4]	± 10% of true value	9020B	CCV ± 10% of true value SOP NO. CORP-WC-0001
	Ending	SM 5320B [4] 450.1 [4]	± 10% of true value	9020B	CCV ± 10% of true value SOP NO. CORP-WC-0001
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	Not Applicable
	Continuing	160.3		--	Not Applicable
	Ending	160.3		--	Not Applicable
Total Suspended Solids (Nonfilterable)	Initial	160.2	This is a gravimetric determination. Calibrate balance before use.	--	Not Applicable
	Continuing	160.2		--	Not Applicable
	Ending	160.2		--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range Follow manufacturer's instructions	--	Not Applicable
	Continuing	180.1	Not Applicable	--	Not Applicable
	Ending	180.1	Not Applicable	--	Not Applicable
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	Not Applicable
	Continuing	160.4		--	Not Applicable
	Ending	160.4		--	Not Applicable
Water Content	Initial	--	Calibrate Balance	--	Calibrate Balance
	Continuing	--	Not Applicable	--	Not Applicable
	Ending	--	Not Applicable	--	Not Applicable
GFAA Metals (excludes Hg)	Initial	200 series	3 levels plus blank ICV ± 10% of true value "r"[3] ≥ 0.995 SOP NO. CORP-MT-0003	7000A series	3 levels plus blank ICV ± 10% of true value "r"[3] ≥ 0.995 SOP NO. CORP-MT-0003
	Continuing	200 series	Every 10 samples ± 10% of true value SOP NO. CORP-MT-0003	7000A series	Every 10 samples ± 20% of true value SOP NO. CORP-MT-0003
	Ending	200 series	± 10% of true value SOP NO. CORP-MT-0003	7000A series	± 20% of true value SOP NO. CORP-MT-0003
	Other	200 series	Annually - Instrument detection limits SOP NO. CORP-MT-0003	7000A	Annually - Instrument detection limits SOP NO. CORP-MT-0003

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
ICAP Metals (excludes Hg)	Initial	200.7	1 level and blank Rerun high calibration standard: verify quantitation at ± 5% of true value,ICV RSD < 3% from replicate (SOP No. CORP-MT-0001)	6010B	1 level and blank Rerun high calibration standard: verify quantitation at ± 5% of true value. ICV RSD < 5% from replicate (SOP No. CORP-MT-0001)
	Continuing	200.7	Every 10 samples ± 5% of true value CCV RSD < 5% from replicate (SOP No. CORP-MT-0001)	6010B	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate (SOP No. CORP-MT-0001)
	Ending	200.7	± 5% of true value CCV RSD < 5% from replicate (SOP No. CORP-MT-0001)	6010B	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate (SOP No. CORP-MT-0001)
	Other	200.7	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see Section 9.8, SOP No. CORP-MT-0001. Annually: ICP interelement correction factors Instrument detection limits	6010B	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see Section 9.8, SOP No. CORP-MT-0001. Annually: ICP interelement correction factors Instrument detection limits
ICP/MS Metals	Initial	200.8	1 level and blank. ICV: ± 10% of true	6020	1 level and blank. ICV: ± 10% of true
	Continuing	200.8	Mid-level calibration standard Every 10 samples. ± 10% of true value	6020	Mid-level calibration standard Every 10 samples. ± 10% of true value
	Ending	200.8	Not specified	6020	Mid-level calibration standard. ± 10% of true value
	Other	200.8	Initial tuning standard deviation ≤ 5% for five replicates; Mass calibration ≤ 0.1 amu from true; Resolution ≤ 0.75 amu full width at 5% peak height; Analyze ICSA and ICSAB at the beginning of each run and every 12 hours	6020	Initial tuning standard deviation ≤ 5% for four replicates; Mass calibration ≤ 0.1 amu from true; Resolution ≤ 0.9 amu full width at 10% peak height; Analyze ICSA and ICSAB at the beginning of each run and every 12 hours

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Mercury by CVAA	Initial	245.1 245.5	5 levels plus blank ICV ± 10% of true value "r"[3] ≥ 0.995 SOP NO. CORP-MT-0005/0007	7470A 7471A	5 levels plus blank ICV ± 10% of true value "r"[3] ≥ 0.995 SOP NO. CORP-MT-0005/0007
	Continuing	245.1 245.5	Daily or every 10 samples, whichever is more frequent ± 20% of true value SOP NO. CORP-MT-0005/0007	7470A 7471A	Every 10 samples ± 20% of true value SOP NO. CORP-MT-0005/0007
	Ending	245.1 245.5	± 20% of true value SOP NO. CORP-MT-0005/0007	7470A 7471A	± 20% of original prepared standard SOP NO. CORP-MT-0005/0007
	Other	245.1 245.5	Annually: - Instrument detection limits	7470A 7471A	Annually - Instrument detection limits

Footnotes

(1) National Pollutant Discharge Elimination System

(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).

(3) "r" = correlation coefficient

(4) Method not listed in 40 CFR Part 136.

TABLE 8.5-8
Summary of Organic Method Calibrations

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Aromatic Volatiles by GC	Initial	602	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. SOP No. CORP-GC-0001
	Continuing	602	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 samples. % D ≤ 15%, gases 20% D. Evaluate per SOP No. CORP-GC-0001 requirements.
	Ending	602	Not Applicable	8021B	Mid-level calibration standard % D ≤ 15%. Evaluate per SOP No. CORP-GC-0001 requirements.
	Other	602	Not Applicable	8021B	Not Applicable
Acrolein &Acrylonitrile by GC	Initial	603	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8031 (Acrylo-nitrile only)	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	603	Analyze QC check sample and evaluate per method requirements	8031 (Acrylo-nitrile only)	Mid-level calibration standard analyzed every 10 samples. Evaluate per method requirements.8
	Ending	603	Not Applicable	8031 (Acrylo-nitrile only)	Mid-level calibration standard Evaluate per method requirements.
	Other	603	Not Applicable	8031 (Acrylo-nitrile only)	Not Applicable

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Dioxins/ Dibenzofurans (LRMS)	Initial	613	3 levels If % RSD <10%, use mean RF. Otherwise calibration curve employed	8280A	5 levels in triplicate % RSD ≤ 15%
	Continuing	613	1 level each working day. % D must be ≤ 15%.	8280A	1 level every 12 hours after window performance mix Standard must have RFs with %D ≤ 30% from initial
	Ending	613	Not Applicable	8280A	Window performance mix
	Other	613	Establish Single Ion Monitoring conditions described in method	8280A	Window mix to set congener windows every 12 hours at beginning of sequence. Isotope ratios in standard must meet criteria in method. Valley between 2,3,7,8-TCDD[3] and 1,2,3,4-TCDD must be ≤ 25% of the 2,3,7,8-TCDD[3] peak height.

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Dioxins/ Dibenzofurans (HRGC/HRMS)	Initial	1613B	5 levels plus window defining solution. %RSD for compounds calibrated by isotope dilution < 20%. %RSD for compounds calibrated by internal standard < 35%.	8290	1 level every 12 hours after window defining solution. RFs with %D ≤ 20% for natives; %D ≤ 30% for labeled compounds from initial
	Continuing	1613B	1 level every 12 hours after window defining solution. Calculated concentrations must be within method acceptance criteria. (1613 Table 6)	8290	1 level: RFs with %D ≤ 20% for natives: %D ≤ 30% for labeled compounds from initial
	Ending	1613B	Not Applicable	--	Not Applicable
	Other	1613B	Isotope ratios in calibration standards must meet criteria in method. Valley between 2,3,7,8-TCDD and all other TCDDs must be ≤ 25% of the 2,3,7,8-TCDD height.	8290	Isotope ratios in standard must meet criteria in method. Valley between 2,3,7,8-TCDD[3] and all other TCDDs must be ≤ 25% of the 2,3,7,8-TCDD height
PCBs by HRGC/HRMS	Initial	1668	5 levels plus window defining solution. %RSD for compounds calibrated by isotope dilution < 20%. %RSD for compounds calibrated by internal standard < 35%.	--	Not Applicable
	Continuing	1668	1 level every 12 hours after window defining solution. Calculated concentrations must be within method acceptance criteria. (1668 Table 6)	--	Not Applicable
	Ending	1668	Not Applicable	--	Not Applicable
	Other	1668	Isotope ratios in calibration standards must meet criteria in method.	--	Not Applicable

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Herbicides by GC	Initial	615[9]	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. SOP No. CORP-GC-0001
	Continuing	615[9]	1 or more calibration standards analyzed daily % D ± 15% of predicted response	8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported. SOP No. CORP-GC-0001
	Ending	615[9]	Not Applicable	8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported. SOP No. CORP-GC-0001
	Other	615[9]	Not Applicable	8151A	Not Applicable
Nitroaromatics by HPLC	Initial	--	Not Applicable	8330	Minimum of 5 levels. Curve should be linear with zero intercept.
	Continuing	--	Not Applicable	8330	Midpoint calibration standard at beginning and after the midpoint of sample run. %D < 15% of predicted response for any analyte quantitated and reported.

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Nitroaromatics by HPLC (continued)	Ending	–	Not Applicable	8330	Midpoint calibration standard. %D: < 15% of predicted response for any analyte quantitated and reported.
	Other	–	Not Applicable	8330	Not Applicable
Polyaromatic Hydrocarbons by GC or HPLC	Initial	610	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8100 8310	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	610	1 or more calibration standards analyzed daily % D ± 15% of predicted response	8100 8310	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	610	Not Applicable	8100 8310	Mid-level calibration standard. % D ± 15% of predicted response for any analyte quantitated and reported.
	Other	610	Not Applicable	8100 8310	Not Applicable
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8082	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP No. CORP-GC-0001)
	Continuing	608	1 or more calibration standards analyzed daily % D ± 15% of predicted response	8081A 8082	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported. (See SOP No. CORP-GC-0001)

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Pesticides/ PCBs by GC (continued)	Ending	608	Not Applicable	8081A 8082	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported. (See SOP No. CORP-GC-0001)
	Other	608	Not Applicable	8081A 8082	Not Applicable
Petroleum Hydrocarbons/ Oil and Grease	Initial	413.1 413.2 418.1	Method 413.1: This is a gravimetric determination. Calibrate balance before use. Method 413.2/418.1: 3 levels plus a blank "r" ≥ 0.995	9070 9071A	This is a gravimetric determination. Calibrate balance before use
	Continuing	413.1 413.2 418.1	Not Applicable	9070 9071A	Not Applicable
	Ending	413.1 413.2 418.1	Not Applicable	9070 9071A	Not Applicable
	Initial	1664[8]	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance SOP No. CORP-WC-0003	--	--
	Continuing	1664[8]	Not Applicable	--	--
	Ending	1664[8]	Not Applicable	--	--
Organophos-phorous Pesticides by GC	Initial	--	Not Applicable	8141A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Organophos-phorous Pesticides by GC (continued)	Continuing	--	Not Applicable	8141A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	Not Applicable	8141A	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	Not Applicable	8141A	Not Applicable
Purgeable Halocarbons by GC	Initial	601	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP No. CORP-GC-0001)
	Continuing	601	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15%, gases 20% D Evaluate per SOP No. CORP-GC-0001 requirements.
	Ending	601	Not Applicable	8021B	Mid-level calibration standard % D <15% Evaluate per SOP No. CORP-GC-0001 requirements.
	Other	601	Not Applicable	8021B	Not Applicable

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Halogenated Volatiles by GC	Initial	--	Not Applicable	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP No. CORP-GC-0001)
	Continuing	--	Not Applicable	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported. (See SOP No. CORP-GC-0001)
	Ending	--	Not Applicable	8021B	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported. (See SOP No. CORP-GC-0001)
	Other	--	Not Applicable	8021B	Not Applicable
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL If % RSD ≤ 35%, use avg RF Otherwise calibration curve employed. SOP No. CORP-MS-0001	8270C	Minimum of 5 levels. % RSD for RF for CCCs[4] < 30% SPCCs[5]: RF > 0.050 SOP No. CORP-MS-0001
	Continuing	625	1 level every 24 hours Aceeptance criteria are found in the metnod and SOP No. CORP-MS-0001	8270C	Mid-level standard every 12 hours (after tuning) %D for CCCs[4] < 20 % between RF from standard and avg RF from initial SPCCs[5]: RF > 0.050 SOP No. CORP-MS-0001

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Semivolatiles (continued)	Ending	625	Not Applicable	8270C	Not Applicable
	Other	625	DFTPP[7] tuning every 24 hours before standard or sample runs. SOP No. CORP-MS-0001	8270C	DFTPP[7] tuning at the beginning of every 12 hour shift. SOP No. CORP-MS-0001
Volatiles	Initial	--	Not Applicable	8260B	Minimum of 5 levels, %RSD for RF for CCCs[4] < 30.0% SPCCs[5]: RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform (See Table 11, SOP No. CORP-MS-0002)

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Volatiles (continued)	Continuing	--	Not Applicable	8260B	Mid-level standard every 12 hours (after tuning) %Drift for CCCs[4] < 20.0% between RF from standard and avg RF from initial SPCCs[5]: RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform SOP No. CORP-MS-0002
	Ending	--	Not Applicable	8260B	Not Applicable
	Other	--	Not Applicable	8260B	BFB[6]tuning at the beginning of every 12 hour shift. SOP No. CORP-MS-0002

Footnotes

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. (SW-846). Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(3) TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin
(4) CCC - Continuing Calibration Compounds
(5) SPCC - System Performance Check Compound
(6) BFB - Bromofluorobenzene
(7) DFTPP - Decafluorotriphenylphosphine
(8) Method 1664 is a proposed only method and has not been promulgated by the EPA.
(9) Method not listed in 40 CFR Part 136.

TABLE 8.5-9
Summary of USEPA Contract Laboratory Program Statement of Work Method Calibrations

Analytical Parameter	Calibration	Method	Requirement
Cyanide, Total	Initial	ILM03.0 ILMO4.0	Minimum 5 levels plus blank "r" ≥ 0.995
	Continuing	ILM03.0 ILMO4.0	1 mid-level every 10 samples ± 15 % of true value
	Ending	ILM03.0 ILMO4.0	± 15 % of true value
	Other	ILM03.0 ILMO4.0	Not Applicable
ICAP (excludes mercury)	Initial	ILM03.0 ILMO4.0	1 level and blank ICV: ± 10% of true
	Continuing	ILM03.0 ILMO4.0	Mid-level calibration standard Every 10 samples ± 10% of true value
	Ending	ILM03.0 ILMO4.0	Mid-level calibration standard ± 10% of true value
	Other	ILM03.0 ILMO4.0	ILMO3.0: ICSA, ICSAB: Analyze at beginning and end or every 8 hours whichever is more frequent ILMO3.0: CRI: Beginning and end of each run, and every 8 hours for all analytes at 2x CRDL or 2x IDL whichever is greater, except for Al, Ba, Ca, Fe, Mg, Na, K ILMO4.0: ICSA, ICSAB: Analyze at beginning and end of run, but not before ICV. Must be analyzed every 20 analytical samples per ICP run. ILMO4.0: CRI: Beginning and end of each run and every 20 analytical samples per ICP run. CRI must be immediately followed by ICS analysis. Quarterly: Instrument detection limits Linear Range Verification Annually: ICP interelement correction factors

TABLE 8.5-9
Summary of USEPA Contract Laboratory Program Statement of Work Method Calibrations (Continued)

Analytical Parameter	Calibration	Method	Requirement
GFAA (excludes Hg)	Initial	ILM03.0 ILMO4.0	Minimum 3 levels plus blank ICV: ± 10%
	Continuing	ILM03.0 ILMO4.0	Every 10 samples ± 10% of true value
	Ending	ILM03.0 ILMO4.0	± 10% of true value
	Other	ILM03.0 ILMO4.0	CRA: Beginning of every analytical run (no acceptance criteria) Quarterly - Instrument detection limits
Mercury (CVAA)	Initial	ILM03.0 ILMO4.0	Minimum 3 levels plus blank "r"(4) ≥ 0.995 ICV: ± 20%
	Continuing	ILM03.0 ILMO4.0	Every 10 samples ± 20% of true value
	Ending	ILM03.0 ILMO4.0	± 20% of true value
	Other	ILM03.0 ILMO4.0	Quarterly - Instrument detection limits ILMO3.0: CRA not required. ILMO4.0: CRA: Beginning of every analytical run (no acceptance criteria)
Pesticides/PCBs	Initial	OLM03.1	3 levels for single component analytes, 1 level for multicomponent analytes RSD must be ≤ 20% except α-BHC and δ-BHC at 25% (allow up to 2 target analytes to be 20% ≤ 30%)
	Continuing	OLM03.1	Instrument Blank and midpoint calibration or PEM every 12 hours % D: ± 25% of predicted response

TABLE 8.5-9
Summary of USEPA Contract Laboratory Program Statement of Work Method Calibrations (Continued)

Analytical Parameter	Calibration	Method	Requirement
Pesticides/PCBs (continued)	Ending	OLM03.1	Instrument Blank and midpoint calibration or PEM
	Other	OLM03.1	Resolution Check Mixture ≥ 60% PEM: ≥ 90% DDT, Endrin breakdown must each be ≤ 20% (≤ 30% combined)
	Initial	OLM01.8	3 levels for single component analytes, 1 level for multicomponent analytes RSD must be ≤20% for each single component target compounds (up to two single components target compounds per column may be > 20.0% but those compounds must have an RSD ≤30.0%.
	Continuing	OLM01.8	Instrument Blank and mid-point calibration standard or PEM every 12 hours Must meet resolution, retention time window, and RPD requirements in method
	Ending	OLM01.8	Instrument Blank and mid-point calibration standard or PEM Must meet resolution retention time window, and RPD requirements in method
PCDD, PCDF	Initial	DFLM01.1	Minimum 5 levels Resolution: 13C12-2378-TCDD and 13C12-1234-TCDD < 25% 123478-HxCDD and 123678-HxCDD ≤ 50% %RSD unlabeled PCDDs/PCDFs and internal standards ≤ 15%
	Continuing	DFLM01.1	Analyze CC3 or CPS solution every 12 hours Must meet ion abundance, S/N, and %D criteria in method
	Ending	DFLM01.1	Analyze CC1 solution at end of 12 hour period Must meet ion abundance and S/N criteria in method
	Other	DFLM01.1	Window Defining Mix: verify switching times

TABLE 8.5-9
Summary of USEPA Contract Laboratory Program Statement of Work Method Calibrations (Continued)

Analytical Parameter	Calibration	Method	Requirement
Semivolatiles by GC/MS	Initial	OLM03.1	5 levels RRF and RSD must meet method criteria
	Continuing	OLM03.1	Mid-level every 12 hours after tuning check %D and RRF must meet minimum criteria
	Ending	OLM03.1	Not Applicable
	Other	OLM03.1	DFTPP tuning at the beginning of every 12 hour shift
	Initial	OLM01.8	5 levels RRF and RSD must meet method criteria
	Continuing	OLM01.8	1 level every 12 hours %D and RRF must meet minimum method criteria
	Ending	OLM01.8	Not Applicable
Volatiles by GC/MS	Initial	OLM03.1	5 levels RRF and RSD must meet method criteria
	Continuing	OLM03.1	Mid-level every 12 hours after tuning check %D and RRF must meet minimum criteria
	Ending	OLM03.1	Not Applicable
	Other	OLM03.1	BFB tuning at the beginning of every 12 hour shift
	Initial	OLM01.8	5 levels RRF and RSD must meet method criteria
	Continuing	OLM01.8	1 level every 12 hours %D and RRF must meet minimum method criteria
	Ending	OLM01.8	Not Applicable
	Other	OLM01.8	BFB tuning at the beginning of every 12 hour shift

TABLE 8.6-1
Precision and Accuracy Measurements

Measurement	Definition
Accuracy	The degree of agreement of a measurement with an accepted reference or true value. The only true or known values in the laboratory are spiked samples. Expressed as laboratory control sample (LCS) percent recovery (% R): $$LCS\ \%Recovery = \frac{X}{t} x\ 100$$ where: X = observed concentration t = concentration of spike added Expressed as matrix spike/matrix spike duplicate (MS/MSD) sample percent recovery (% R): $$MS/MSD\ \%Recovery = \frac{X_s - X}{t} x\ 100$$ where: X_S = observed concentration in spiked sample X = observed concentration in unspiked sample t = concentration of spike added
Precision	The measure of analytical reproducibility of two values. Expressed as the relative percent difference (RPD) of two values. $$RPD = \left[\frac{\lvert X_1 - X_2 \rvert}{\left(\frac{X_1 + X_2}{2}\right)}\right] x\ 100$$ where: X_1 = first observed concentration X_2 = second observed concentration

TABLE 8.6-1
Precision and Accuracy Measurements
(Continued)

Measurement	Definition
Arithmetic mean	The average of a set of values. $$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$ where: $\bar{x}$ = the mean x_i = the ith data value n = number of data values
Standard Deviation	A measure of the random (probable) error associated with a single measurement within a data set. $$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$$ where: s = sample standard deviation $\bar{x}$ = the mean x_i = the ith data value n = number of data values
Quality Control Chart	A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.
ACCURACY	
Upper Control Limit (UCL)	$UCL = \bar{x} + 3s$
Upper Warning Limit (UWL)	$UWL = \bar{x} + 2s$
Lower Warning Limit (LWL)	$LWL = \bar{x} - 2s$
Lower Control Limit (LCL)	$LCL = \bar{x} - 3s$
PRECISION	
RPD	Zero to (mean RPD + 3s)

TABLE 8.11-1
Instrument Maintenance Schedule
Ion Chromatograph[1]

As Needed	Daily	Weekly	Monthly	Semi-annually
Clean micromembrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.	Lubricate left hand piston.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.	Clean conductivity cell.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.			Check conductivity cell for calibration.
De-gas pump head when flow is erratic.	Check conductivity meter.			

TABLE 8.11-2
Instrument Maintenance Schedule
LACHAT Auto Analyzer[1]

As Needed	Daily	Monthly	Semi-annually	Annually
Prepare fresh reagents.	Check detector. Clean detector cell and make sure there are no trapped bubbles in detector cell. Check Valves Check Reference source	Replace tubing.	Lubricate pump roller.	Clean pump rollers with steel wool and lubricate.
	Check peristaltic tubing. Check sampler Check auto diluter	Clean pump, diluter, and XYZ Sampler.		
	Clean sample probe shaft.			

TABLE 8.11-3
Instrument Maintenance Schedule
Total Organic Halide Analyzer[1]

Daily	As Needed
Check electrodes for damage, polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace o-rings and seals.

TABLE 8.11-4
Instrument Maintenance Schedule
High Pressure Liquid Chromatograph[1]

Daily	As Needed
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

TABLE 8.11-5
Instrument Maintenance Schedule
Flame Atomic Absorption Spectroscopy[1]

Daily	Monthly	As Needed
Verify proper safety precautions are working.	Clean all filters and fans.	Check drain receptacle.
Verify gas box operates properly and safely.	Change capillary tubing	Check background corrector for alignment.
Verify sensitivity using elements in UV/VIS spectrum.	Clean optical windows	Clean burner head.
		Clean nebulizer.
		Clean spray chamber.
		Check sample introduction O-rings.

TABLE 8.11-6
Instrument Maintenance Schedule
Inductively Coupled Argon Plasma/Mass Spectrometry (ICAP/MS)[1]

Daily	Weekly	Monthly	Quarterly	Annually	As Needed
Check sample waste container level.	Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing.	Clean all filters and fans.	Replace oil in roughing pumps.	Replace oil in turbo-molecular pump.	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics, CEM, deflector voltage.
Check quartz torch condition.	Check condition of sampler and skimmer cones.	Check recirculator water level.			
Measure quartz torch for proper alignment.	Check and drain oil mist eliminator on roughing pumps.				
Clean spray chamber and nebulizer.					
Check oil level of roughing pumps.					

TABLE 8.11-7
Instrument Maintenance Schedule
ICP[1]

Daily	Monthly or As Needed	Semi-annually	Annually
Check gases Check that argon tank pressure is 50-60 psi and that a spare tank is available. Check aspiration tubing	Clean plasma torch assembly to remove accumulated deposits.	Change vacuum pump oil.	Notify manufacturer service engineer for scheduled preventive maintenance service.
Check vacuum pump gage. (<10 millitorr)	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance.	Replace coolant water filter. (may require more or less frequently depending on the quality of water)	
Check that cooling water supply system is full and drain bottle is not full. Also that drain tubing is clear, tight fitting and has few bends.	Clean filters on back of power unit to remove dust.		
Check that nebulizer is not clogged.	Replace when needed: peristaltic pump tubing sample capillary tubing autosampler sipper probe		
Check that capillary tubing is clean and in good condition.	Check yttrium position. Check O-rings Clean/lubricate pump rollers.		
Check that peristaltic pump windings are secure.			
Check that high voltage switch is on.			
Check that exhaust screens are clean.			
Check that torch, glassware, aerosol injector tube, bonnet are clean.			

TABLE 8.11-8
Instrument Maintenance Schedule
Graphite Furnace Atomic Absorption[1]

Daily	Weekly	Monthly	Semi-annually	Annually
Check gas lines and gas supply.	Clean optical windows.	Check coolant level in cooling unit. Add coolant if error message appears.	Change graphite contacts	Notify manufacturer service engineer to clean optics. Check optics
Clean contact cylinders.				
Check tubes and platform; replace if corroded, faking, or if low absorbance results. Check autosampler tubing and alignment. Flush autosampler tubing				
PE4100ZL: clean fume extraction tip, replace fume extraction filter and H_2O trap.				
As needed, trim sampling capillary.				
Check drain lines and waste containers; empty as needed.				
Check acid rinse containers; fill as needed.				

TABLE 8.11-9
Instrument Maintenance Schedule
Cold Vapor Atomic Absorption (Leeman PS 200)[1]

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp.
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

TABLE 8.11-10
Instrument Maintenance Schedule
Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	As Needed	Monthly
Clean aspirator by flushing with DI water.	Change source lamp	Clean cell in aqua regia.
Check tubing and replace if needed.		Clean aspirator in aqua regia.
Change silica gel in drying tube.		Clean windows with methanol.
Check argon gas supply.		
Adjust lamp.		

TABLE 8.11-11
Instrument Maintenance Schedule
Gas Chromatograph[1]

Daily	As Needed	Quarterly/Semi-annually/Annually
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Quarterly ELCD: change-roughing resin, clean cell assembly. Quarterly FID: clean detector
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.	Semi-annually ECD: perform wipe test.
Check inlets, septa. Replace septum Clean injector port		Annually ELCD: change finishing resin, clean solvent filter. Annually FID: Replace flame tip ECD: detector cleaning and re-foiling, every five years or whenever loss of sensitivity, or erratic response or failing resolution is observed.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).	
Check reactor temperature of electrolytic conductivity detector.	Replace or repair flow controller if constant gas flow cannot be maintained.	
	Replace fuse.	
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Reactivate external carrier gas dryers.	
Clip column leader	Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. NPD: clean/replace collector assembly. PID: clean lamp window monthly or replace as needed, replace seals. ELCD: check solvent flow weekly, change reaction tube, replace solvent, change reaction gas, clean/replace Teflon® transfer line. ECD: follow manufacturers suggested maintenance schedule	
	Reactivate flow controller filter dryers when presence of moisture is suspected.	

TABLE 8.11-11
Maintenance Schedule
Gas Chromatograph[1]
(Continued)

Daily	As Needed	Quarterly/Semi-annually/Annually
GC (continued)	HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.	
	Purge & trap devices: periodic leak checks quarterly, replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), clean glassware. Clean sparger weekly. Check purge flow monthly. Bake trap as needed to correct for high background. Change trap annually, or as needed whenever loss of sensitivity, or erratic response or failing resolution is observed.	
	Purge & trap autosamplers: leak check system, clean sample lines, valves. PTA-30 autosampler also requires cleaning the syringes, frits, valves, and probe needles, adjustment of micro switches, replacement of Teflon® valve, and lubrication of components.	

TABLE 8.11-12
Instrument Maintenance Schedule
Mass Spectrometer[1]

Daily	Weekly	As Needed[2]	Quarterly	Semi-Annually	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check ion source and analyzer (clean, replace parts as needed)		Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	Clean rods	
Check inlets, septa.		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump. Relubricate the turbomolecular pump-bearing wick.		
Check baseline level.		Repair/replace jet separator.			
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.			

TABLE 8.11-13
Instrument Maintenance Schedule
TRAACS 800 Auto Analyzer [1]

As Needed	Daily	Monthly	Semi-annually	Annually
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)	Change all pump tubes (or after 200 hours of pumping time)	(or after 1000 hours of pumping time)	Lightly lubricate the Linear Sample Rails (use semi-fluid lubricant)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)	Clean sample probe shaft	Replace pump platens	Replace colorimeter lamp (or after 2500 hours of use)

TABLE 8.11-14
Instrument Maintenance Schedule
Sonicator [1]

Daily	As Needed
Daily when used: Inspect probe tips for inconsistencies (etching/pitting).	Replace probe tip.
	Disassemble and clean sonicator probe tips.
	Tune sonicator assembly.

TABLE 8.11-15
Instrument Maintenance Schedule
Analytical/Top Loading Balances[1]

Daily	Annually
Check using Class S-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration.

TABLE 8.11-16
Instrument Maintenance Schedule
Refrigerators/Walk-in Coolers[1]

Daily	As Needed
Temperatures checked and logged.	Refrigerant system and electronics serviced.

TABLE 8.11-17
Instrument Maintenance Schedule
Ovens[1]

Daily	As Needed
Temperatures checked and logged.	Electronics serviced.

TABLE 8.11-18
Instrument Maintenance Schedule
Specific Digital Ion Analyzer[1]

Daily	As Needed
Daily when used: Calibrate with check standards. Inspect electrode daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use.	Electronics serviced.

TABLE 8.11-19
Instrument Maintenance Schedule
Turbidimeter[1]

Daily	Monthly	As Needed
Daily when used: Adjust linearity on varying levels of NTU standards. Standardize with NTU standards. Inspect cells.	Clean instrument housing	Electronics serviced.

TABLE 8.11-20
Instrument Maintenance Schedule
Dissolved Oxygen Meter[1]

Daily	As Needed
Daily when used: Calibrate with check standards. Check probe membrane for deterioration Clean and replace membrane with HCl solution.	Electronics serviced.

TABLE 8.11-21
Instrument Maintenance Schedule
Conductance Meter[1]

Daily	As Needed
Daily when used: Check probe and cables. Standardize with KCl. Inspect conductivity cell.	Electronics serviced.

TABLE 8.11-22
Instrument Maintenance Schedule
Chemical Oxygen Demand (COD) Reactor

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

TABLE 8.11-23
Instrument Maintenance Schedule
Spectrophotometer[1]

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens.	Check the zero %T adjustment.	Clean windows	Oil bearings.
	Clean sample compartment		Perform wavelength calibration at 530 nm.
	Clean cuvettes		Replace lamp annually or when erratic response is observed.
			Clean and align optics.

TABLE 8.11-24
Instrument Maintenance Schedule
pH Meter[1]

As Needed	Daily
Clean electrode.	Inspect electrode. Verify electrodes are properly connected and filled.
Refill reference electrode.	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer.

TABLE 8.11-25
Instrument Maintenance Schedule
Fourier Transform Infrared Spectrometry (FTIR)[1]

Check desiccant every 3 months.
Check KBr window every 3 months.

TABLE 8.11-26
Instrument Maintenance Schedule
Radiological Analysis Equipment[1]

Instrument	Items Checked/Service	Minimum Frequency
Alpha Proportional	Check gas flow	Daily
	Clean sample tray	Weekly
	Check bubbler oil level	Monthly
Beta Proportional	Check gas flow	Daily
	Clean sample holders	Weekly
Liquid Scintillation	Clean sample changer	Weekly
	Check condensate trays	Weekly
	Check air filters	Monthly
Quad aß Proportional	Check gas flow	Daily
	Clean sample holders	Weekly
Gamma Spectroscopy	Check LN_2 level	Bi-weekly
	Replace plastic liner	Weekly
Alpha Spectroscopy	Clean sample holder	As needed
	Change vacuum pump oil	Every six months
LIPA	Clean sample changer	Weekly
	Check laser dye performance	Weekly
Benzene Synthesizer	Check gas tubes	Weekly
	Clean instrument	Monthly
Electrolytic Enrichment	Check electrical leads	Monthly
	Clean system	Monthly
Fluorometer	Clean sample holder	Weekly

TABLE 8.11-27
Instrument Maintenance Schedule
Total Organic Carbon Analyzer (OI 7000)

Daily	As Needed	Weekly	Monthly	Semi-Annually
Check: Oxygen supply Persulfate supply Acid supply Carrier gas flow rate (~ 150 cc/min) IR millivolts for stability (after 30 min. warm-up) Reagent reservoirs	Check injection port septum after 50-200 runs. Tube end-fitting connections after 100 hours or use. Indicating drying tube. NDIR zero, after 100 hours of use. Sample pump, after 2000 hours for use. Digestion vessel/condensation chamber, after 2000 hours of use. Permeation tube, after 2000 hours of use. NDIR cell, after 2000 hours of use.	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test	Change pump tubing

Footnotes to Preventive Maintenance Tables

(1) Refer to manufacturer's instructions for each instrument to perform maintenance operations.
(2) Also see Table 8.11-11 for applicable "As Needed" GC maintenance.

TABLE 8.11-28
Instrument Maintenance Schedule
APCI/ESI LC/MS/MS

Daily	As Needed	Semi-annually	Annually
Check helium sparge supply for adequate pressure	Oil autosampler when it is noisy or picking up the tray	Replace rough-pump oil	Replace turbo-pump oil
Check solvent reservoirs for sufficient level of solvent	Change pump seals		Vacuum system components including fans and fan covers
Verify that pump is primed, operating pulse free	Change filters in autosampler		
Check needle wash reservoir for sufficient solvent	Replace column if excessive pressure or poor performance		
Verify capillary heater temperature	Rinse capillary with MeOH		
Verify vaporizer heater temperature	Clear capillary if clogged		
Verify manifold heater temperature	Rinse and clean corona needle		
Verify manifold pressure (~5 x 10-6)	Replace sample inlet tube in APCI (10.1 cm)		
Verify fore-pump pressure (~30 to 200mtorr)	Replace fused silica tube in ESI interface		
Verify rough pump and turbo-pump oil levels	Clean lenses		
Verify nitrogen pressure for auxiliary and sheath gasses	Clean skimmer		
Verify that corona and multiplier are functioning			

TABLE 8.11-29
Instrument Maintenance Schedule
Digestion Block

Annually
Check temperature with NIST thermometer

TABLE 8.11-30
Instrument Maintenance Schedule
Flash Point Tester

Daily	As Needed
Check tubing. Clean sample cup each use.	Check thermometer against NIST thermometer, when used.
Check gas.	
Clean flash assembly	
Check stirrer	

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Index

Index (continued)

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Index (continued)

Page/Location

Accuracy (See Precision and Accuracy Measurements)

Anomaly (See Nonconformance)

Assessment (Quality) 67, 88
- Audit (See Audit)
- Data Accuracy (See Precision and Accuracy Measurements)
- Data Completeness 45
- Data Precision (See Precision and Accuracy Measurements)
- Data Quality Objectives 44
- Management Review of the QMS 85
- Quality Reports to Management 84

Audit (Quality Assurance) 80
- Data Audit 83
- Findings 82
- Areas Needing Improvement 82
- Performance Audit 81
- Report 84
- Spot Assessment 83
- Systems Audit (Internal) 81
- Systems Audit (External) 83

Benchmarking 88

Calibration
- Contract Laboratory Program (CLP) Method Calibrations
  - Cyanide, Total ..... Table 8.5-9
  - GFAA ..... Table 8.5-9
  - ICAP ..... Table 8.5-9
  - Mercury (CVAA) ..... Table 8.5-9
  - PCDD/PCDF ..... Table 8.5-9
  - Pesticides/PCBs ..... Table 8.5-9
  - Semivolatiles ..... Table 8.5-9
  - Volatiles ..... Table 8.5-9

Index (continued)

Page/Location

Calibration (continued)
Criteria ..... 63-64, Tables 8.5-7, 8.5-8, 8.5-9
Failure 66
Inorganic Method Calibrations
Acidity ..... Table 8.5-7
Alkalinity ..... Table 8.5-7
Ammonia ..... Table 8.5-7
Biochemical Oxygen Demand (BOD) ..... Table 8.5-7
Bromide ..... Table 8.5-7
Chemical Oxygen Demand (COD) ..... Table 8.5-7
Chloride ..... Table 8.5-7
Chlorine, Residual ..... Table 8.5-7
Chromium (Hexavalent) Cr^{+6} ..... Table 8.5-7
Color ..... Table 8.5-7
Conductivity ..... Table 8.5-7
Cyanide (Amenable) ..... Table 8.5-7
Cyanide (Total) ..... Table 8.5-7
Flashpoint ..... Table 8.5-7
Fluoride ..... Table 8.5-7
Hardness ..... Table 8.5-7
Iodide ..... Table 8.5-7
Iron, Phenanthroline Method ..... Table 8.5-7
Methylene Blue Active Substances (MBAS) ..... Table 8.5-7
Metals ..... Table 8.5-7
Graphite Furnace AA ..... Table 8.5-7
ICAP ..... Table 8.5-7
ICP/MS ..... Table 8.5-7
Mercury (CVAA) ..... Table 8.5-7
Nitrate ..... Table 8.5-7
Nitrite ..... Table 8.5-7
Nitrate-Nitrite ..... Table 8.5-7
Odor ..... Table 8.5-7
pH ..... Table 8.5-7
Phenolics ..... Table 8.5-7

Index (continued)

Page/Location

Calibration (continued)

Phosphate ..... Table 8.5-7
Phosphorous (Total and Orthophosphate) ..... Table 8.5-7
Reactivity ..... Table 8.5-6
Reactivity ..... Table 8.5-7
Settleable Solids ..... Table 8.5-7
Silica, Dissolved ..... Table 8.5-7
Specific Conductance ..... Table 8.5-7
Sulfate ..... Table 8.5-7
Sulfide ..... Table 8.5-7
Sulfite ..... Table 8.5-7
Temperature ..... Table 8.5-7
Total Dissolved Solids ..... Table 8.5-7
Total Kjeldahl Nitrogen (TKN) ..... Table 8.5-7
Total Organic Carbon (TOC) ..... Table 8.5-7
Total Organic Halides (TOX) ..... Table 8.5-7
Total Solids ..... Table 8.5-7
Total Suspended Solids ..... Table 8.5-7
Total Volatile Solids ..... Table 8.5-7
Turbidity ..... Table 8.5-7
Water Content ..... Table 8.5-7

Organic (Method Calibrations)

Aromatic Volatiles by GC ..... Table 8.5-8
Dioxins/Dibenzofurans by HRGC/HRMS ..... Table 8.5-8
Dioxins/Dibenzofurans by HRGC/LRMS ..... Table 8.5-8
Halogenated Volatiles by GC ..... Table 8.5-8
Herbicides by GC ..... Table 8.5-8
Nitroaromatics by HPLC ..... Table 8.5-8
Organophosphorous Pesticides by GC ..... Table 8.5-8
Polyaromatic Hydrocarbons (PAHs) by GC or HPLC ..... Table 8.5-8
Pesticides/PCBs by GC ..... Table 8.5-8
Petroleum Hydrocarbons/Oil and Grease ..... Table 8.5-8
Purgeable Halocarbons by GC ..... Table 8.5-8
Acrolein and Acrylonitrile ..... Table 8.5-8

Index (continued)

Page/Location

Calibration (continued)
- Semivolatiles ..... Table 8.5-8
- Volatiles ..... Table 8.5-8

Operational 66
Periodic ..... 65, Table 8.5-6
- Balance ..... Table 8.5-6
- Freezer ..... Table 8.5-6
- Graduated Glassware ..... Table 8.5-6
- Micropipettor ..... Table 8.5-6
- Oven ..... Table 8.5-6
- Refrigerator ..... Table 8.5-6
- Syringe ..... Table 8.5-6
- Thermometer ..... Table 8.5-6
- Volumetric Glassware ..... Table 8.5-6

Procedures 63-64
Records 27

Chain-of-Custody
- Chain-of-Custody Form 56
- Internal Chain-of-Custody 62
- Return Chain-of-Custody 63

Chemical Storage 76

Client Inquiries and Complaints 84

Comparability (Data) 46

Completeness (Data) 45, 67

Computer (See also Software)
- Backup 31
- Hardware 31
- Security 31
- Viruses 33

Index (continued)

Page/Location

Condition Upon Receipt (See Nonconformance)

Contingency Planning 38

Control Chart ..... Table 8.6-1

Corrective Action 38

Customer Service Team 34

Data
- Assessment 67
- Collection 35
- Electronic Data Transfer 73
- Qualifiers 70, 73
- Recording Procedures 68
- Reduction 68, 69
- Reporting 72
- Reports 72
- Review 68
- Statistical Evaluation of Data 67
- Validation 74
- Verbal Results 73
- Verification 68-71

Data Quality Objectives 44

Deficiency (See Nonconformance)

Deliverables 73

Document
- Approval ..... 25, Table 5.1-1
- Control 25
- Controlled 25

Index (continued)

Page/Location

Document (continued)
- Distribution 25
- Quality ..... 12, 25, Table 5.1-1, Table 5.2-1
- Review ..... 25, Table 5.2-1
- Revision ..... 25, Table 5.2-1
- Confidentiality 29
- Storage, Retention and Disposal of Records 29

Ethics 9

Facility Security 77

Gas, compressed (See Internal QC Requirements)

Holding Times (See Sample Containers, Preservatives, and Holding Times)

Instrument Maintenance (See Maintenance)

Internal QC Requirements
- Air (Compressed) 76
- Gases (Compressed) 76
- Glassware 76
- Water 76

Key Result Indicators 88-89

Maintenance
- Equipment 74-75
- Facility 75
- Preventative Maintenance 74
- Instrument Maintenance 74-75
- Instrument Maintenance Schedules
  - APCI/ESI LC/MS/MS ..... Table 8.11-28
  - Balances (Analytical, Top Loading) ..... Table 8.11-15
  - Chemical Oxygen Demand (COD) Reactor ..... Table 8.11-22

Index (continued)

Page/Location

Maintenance (continued)

Cold Vapor Atomic Absorption (CVAA) - Leeman PS 200 ..... Table 8.11-9
Cold Vapor Atomic Absorption (CVAA) - PE 5000 ..... Table 8.11-10
Conductance Meter ..... Table 8.11-21
Digestion Block ..... Table 8.11-29
Dissolved Oxygen Meter ..... Table 8.11-20
Flame Atomic Absorption (Flame AA) ..... Table 8.11-5
Flash Point Tester ..... Table 8.11-30
Fourier Transform Infrared Spectrometry (FTIR) ..... Table 8.11-25
Gas Chromatograph (GC) ..... Table 8.11-11
Graphite Furnace Atomic Absorption (GFAA) ..... Table 8.11-8
High Pressure Liquid Chromatography (HPLC) ..... Table 8.11-4
ICAP/MS ..... Table 8.11-6
ICP ..... Table 8.11-7
Ion Chromatograph (IC) ..... Table 8.11-1
LACHAT Auto Analyzer ..... Table 8.11-2
Mass Spectrometer (MS) ..... Table 8.11-12
Ovens ..... Table 8.11-17
pH Meter ..... Table 8.11-24
Radiological Analysis Equipment ..... Table 8.11-26
Refrigerator ..... Table 8.11-16
Sonicator ..... Table 8.11-14
Specific Digital Ion Analyzer ..... Table 8.11-18
Spectrophotometer ..... Table 8.11-23
Total Organic Carbon (TOC) Analyzer (OI 7000) ..... Table 8.11-27
Total Organic Halide (TOX) Analyzer ..... Table 8.11-3
TRAACS 800 Auto Analyzer ..... Table 8.11-13
Turbidimeter ..... Table 8.11-19
Walk-in Cooler ..... Table 8.11-16

Service Agreements 74

Method

Analytical ..... 43, Table 8.2-1
Modifications 44

Index (continued)

Page/Location

Method Detection Limit (See Reporting Limit)

Method of Standard Additions 53

Nonconformance 79
- Anomaly 79
- Condition Upon Receipt Report (CUR) ..... 59, Figure 8.5-2
- Corrective Action 79
- Deficiency 79
- Nonconformance Log 80
- Nonconformance Memo 80
- Responsibilities 80

Organization
- Chart (Also see Section 1 of the Facility-Specific Appendix C and Appendix A) ..... Figure 1.3-1
- Structure 2

Orientation (See Training)

Performance Evaluation Samples (Also See Section 6 of the Facility-Specific Appendix C) 81

Practical Quantitation Limit (See Reporting Limit)

Precision and Accuracy Measurements
- Accuracy ..... 45, Table 8.6-1
- Arithmetic Mean ..... Table 8.6-1
- Laboratory QC Measurements ..... Table 8.4-3
- Lower Control Limit (LCL) ..... Table 8.6-1
- Lower Warning Limit (LWL) ..... Table 8.6-1
- Matrix QC Measurements ..... Table 8.4-4
- Precision ..... 45, Table 8.6-1
- Standard Deviation ..... Table 8.6-1
- Upper Warning Limit (UWL) ..... Table 8.6-1
- Upper Control Limit (UCL) ..... Table 8.6-1

Index (continued)

Page/Location

Preservatives (See Sample Containers, Preservatives, and Holding Times)

Preventive Maintenance (See Maintenance)

Project Planning 35

Procedures (Standardization of) 87

Procurement (Also see Vendor) 19
- Procedures 21
- Reference Materials (See Standards)
- Role of Corporate Director of Contracts 21-21
- Role of Quanterra® Purchasing 21
- Selection of Vendors 19-20
- Subcontract Laboratory Service 23

Project Planning 35

Project-Specific Reporting Limit (See Reporting Limit)

Qualification, Associate (See Training)

Quality Assurance 11-12

Quality Assurance Management Plan (QAMP) 13

Quality Assurance Program or Project Plan (QAPP) 13-14

Quality Assurance Summary (QAS) 14

Quality Control Samples 48
- Contract Laboratory Program (CLP) QC Samples
  - Cyanide, Total ..... Table 8.4-7
  - GFAA ..... Table 8.4-7
  - ICAP ..... Table 8.4-7

Index (continued)

Page/Location

Quality Control Samples (continued)
- Mercury (CVAA) ..... Table 8.4-7
- PCDD/PCDF ..... Table 8.4-7
- Pesticides/PCBs ..... Table 8.4-7
- Semivolatiles by GC/MS ..... Table 8.4-7
- Volatiles by GC/MS ..... Table 8.4-7

Field ..... 48, Table 8.4-1
- Collocated Sample ..... 49, Table 8.4-1
- Field Blank ..... 49, Table 8.4-1
- Field Duplicate ..... 49, Table 8.4-1
- Field Matrix Spike ..... 49, Table 8.4-1
- Rinsate Blank ..... 49, Table 8.4-1
- Split Sample ..... 50, Table 8.4-1
- Trip Blank ..... 49, Table 8.4-1

Inorganic (QC Samples)
- Acidity ..... Table 8.4-5
- Alkalinity ..... Table 8.4-5
- Ammonia ..... Table 8.4-5
- Ammonia (TKN) ..... Table 8.4-5
- Biochemical Oxygen Demand (BOD) ..... Table 8.4-5
- Bromide ..... Table 8.4-5
- Chemical Oxygen Demand (COD) ..... Table 8.4-5
- Chloride ..... Table 8.4-5
- Chlorine, Residual ..... Table 8.4-5
- Chromium (Hexavalent) Cr^{+6} ..... Table 8.4-5
- Color ..... Table 8.4-5
- Conductivity ..... Table 8.4-5
- Cyanide (Amenable) ..... Table 8.4-5
- Cyanide (Total) ..... Table 8.4-5
- Flashpoint ..... Table 8.4-5
- Fluoride ..... Table 8.4-5
- Hardness ..... Table 8.4-5
- Iodide ..... Table 8.4-5
- Iron, Phenanthroline Method ..... Table 8.4-5
- Methylene Blue Active Substances (MBAS) ..... Table 8.4-5

Index (continued)

Page/Location

Quality Control Samples (continued)

Metals ..... Table 8.4-5
Flame AA ..... Table 8.4-5
Graphite Furnace AA ..... Table 8.4-5
ICP ..... Table 8.4-5
ICP/MS ..... Table 8.4-5
Mercury (CVAA) ..... Table 8.4-5
Nitrate ..... Table 8.4-5
Nitrite ..... Table 8.4-5
Nitrate-Nitrite ..... Table 8.4-5
Odor ..... Table 8.4-5
pH ..... Table 8.4-5
Phenolics ..... Table 8.4-5
Phosphate ..... Table 8.4-5
Phosphorous (Total and Orthophosphate) ..... Table 8.4-5
Reactivity (Cyanide and Sulfide) ..... Table 8.4-5
Silica, Dissolved ..... Table 8.4-5
Solids ..... Table 8.4-5
Specific Conductance ..... Table 8.4-5
Sulfate ..... Table 8.4-5
Sulfide ..... Table 8.4-5
Sulfite ..... Table 8.4-5
Temperature ..... Table 8.4-5
Total Organic Carbon (TOC) ..... Table 8.4-5
Total Organic Halides (TOX) ..... Table 8.4-5
Turbidity ..... Table 8.4-5
Water Content ..... Table 8.4-5

Laboratory QC Samples ..... 50, Table 8.4-2, Table 8.4-3
Analytical Spike ..... 53, Table 8.4-2
Duplicate ..... 53, Table 8.4-2, Table 8.4-4
Instrument/Calibration Blank ..... 51, Table 8.4-2, Table 8.4-3
Interference Check Sample ..... 53, Table 8.4-2
Internal Standard ..... 53, Table 8.4-2
Laboratory Control Sample ..... 52, Table 8.4-2, Table 8.4-3
Laboratory Performance QC Samples ..... Table 8.4-3

Index (continued)

Page/Location

Quality Control Samples (continued)
Matrix Spike ..... 52, Table 8.4-2, Table 8.4-4
Matrix Spike Duplicate ..... 52, Table 8.4-2, Table 8.4-4
Method Blank ..... 51, Table 8.4-2, Table 8.4-3
Method of Standard Additions 53
Post Digestion Spike (See Analytical Spike)
Surrogate ..... 53, Table 8.4-2
Matrix QC Samples ..... Table 8.4-4
Organic QC Samples
Aromatic Volatiles by GC ..... Table 8.4-6
Dioxins/Dibenzofurans by HRGC/HRMS ..... Table 8.4-6
Dioxins/Dibenzofurans by HRGC/LRMS ..... Table 8.4-6
Halogenated Volatiles by GC ..... Table 8.4-6
Herbicides ..... Table 8.4-6
Nitroaromatics by HPLC ..... Table 8.4-6
Organophosphorous Pesticides ..... Table 8.4-6
Polyaromatic Hydrocarbons (PAHs) by HPLC and GC ..... Table 8.4-6
Pesticides/PCBs ..... Table 8.4-6
Petroleum Hydrocarbons ..... Table 8.4-6
Purgeable Halocarbons by GC ..... Table 8.4-6
Acrolein and Acrylonitrile ..... Table 8.4-6
Semivolatiles ..... Table 8.4-6
Volatiles by GC/MS ..... Table 8.4-6
Radiological QC Samples
(Also See Section 7 of the Facility-Specific Appendix C, where applicable) 54
Carrier 54
Tracer 54
Yield Monitors ..... 54, Table 8.4-2

Quality Document (See Document, Quality)

Quality Improvement 87

Quality Management System (QMS) 1

Index (continued)

Page/Location

Quality Organization 2

Quality Policy Document 11

Quality-Related Item (QRI) ..... 20, Table 4.2-1

Quality Report to Management 84

Radioisotope ..... 54, Table 8.5-3

Record
- Attendance (See Training, Attendance Records)
- Disposal 29
- Project 28
- Management 27
- Quality 27
- Retention 29

Reference Material (See Standards)

Reference Standards (See Standards)

Reporting Limit 47
- Maximum Contaminant Level (MCL) 47
- Method Detection Limit (MDL) 46
- Minimum Detectable Concentration 48
- Project-Specific Reporting Limit (PSRL) 47
- Radiochemical 47-48

Resume (Associate) 17

Index (continued)

Page/Location

Sample Containers, Preservations, and Holding Times

Contract Laboratory Program (CLP)

Cyanide, Total and Amenable to Chlorination ..... Table 8.5-4
PCDD/PCDF ..... Table 8.5-4
GFAA ..... Table 8.5-4
ICAP ..... Table 8.5-4
Mercury (CVAA) ..... Table 8.5-4
Pesticides/PCBs ..... Table 8.5-4
Semivolatiles ..... Table 8.5-4
Volatiles ..... Table 8.5-4

Inorganic (Sample Containers, Preservations, and Holding Times)

Acidity ..... Table 8.5-1
Alkalinity ..... Table 8.5-1
Ammonia ..... Table 8.5-1
Biochemical Oxygen Demand (BOD) ..... Table 8.5-1
Bromide ..... Table 8.5-1
Chemical Oxygen Demand (COD) ..... Table 8.5-1
Chloride ..... Table 8.5-1
Chlorine, Residual ..... Table 8.5-1
Chromium (Hexavalent) Cr^{+6} ..... Table 8.5-1
Color ..... Table 8.5-1
Conductivity ..... Table 8.5-1
Cyanide (Amenable) ..... Table 8.5-1
Cyanide (Total) ..... Table 8.5-1
Flashpoint (Ignitability) ..... Table 8.5-1
Fluoride ..... Table 8.5-1
Hardness (Total) ..... Table 8.5-1
Iodide ..... Table 8.5-1
Iron, Phenanthroline Method ..... Table 8.5-1
Methylene Blue Active Substances (MBAS) ..... Table 8.5-1
Metals ..... Table 8.5-1
Mercury (CVAA) ..... Table 8.5-1
Nitrate ..... Table 8.5-1
Nitrite ..... Table 8.5-1

Index (continued)

Page/Location

Sample Containers, Preservations, and Holding Times (continued)

Nitrate-Nitrite ..... Table 8.5-1
Odor ..... Table 8.5-1
Orthophosphate ..... Table 8.5-1
pH ..... Table 8.5-1
Phenolics ..... Table 8.5-1
Phosphate ..... Table 8.5-1
Phosphorous (Total) ..... Table 8.5-1
Reactivity (Cyanide and Sulfide) ..... Table 8.5-1
Settleable Solids ..... Table 8.5-1
Silica, Dissolved ..... Table 8.5-1
Specific Conductance ..... Table 8.5-1
Sulfate ..... Table 8.5-1
Sulfide ..... Table 8.5-1
Sulfite ..... Table 8.5-1
Temperature ..... Table 8.5-1
Total Dissolved Solids ..... Table 8.5-1
Total Kjeldahl Nitrogen (TKN) ..... Table 8.5-1
Total Organic Carbon (TOC) ..... Table 8.5-1
Total Organic Halides (TOX) ..... Table 8.5-1
Total Solids ..... Table 8.5-1
Total Suspended Solids ..... Table 8.5-1
Total Volatile Solids ..... Table 8.5-1
Turbidity ..... Table 8.5-1
Water Content ..... Table 8.5-1

Organic

Aromatic Volatiles ..... Table 8.5-2
Dioxins/Dibenzofurans ..... Table 8.5-2
Halogenated Volatiles ..... Table 8.5-2
Herbicides ..... Table 8.5-2
Nitroaromatics ..... Table 8.5-2
Organophosphorous Pesticides ..... Table 8.5-2
Pesticides/PCBs ..... Table 8.5-2
Petroleum Hydrocarbons/Oil and Grease ..... Table 8.5-2

Index (continued)

Page/Location

Sample Containers, Preservations, and Holding Times (continued)

Polyaromatic Hydrocarbons (PAH) by GC and HPLC ..... Table 8.5-2
Purgeable Halocarbons ..... Table 8.5-2
Acrolein and Acrylonitrile ..... Table 8.5-2
Semivolatiles ..... Table 8.5-2
Volatiles ..... Table 8.5-2

Radiological

Alpha/Beta (Gross) ..... Table 8.5-3
Americium-241 ..... Table 8.5-3
Carbon-14 ..... Table 8.5-3
Calcium-45 ..... Table 8.5-3
Curium-242 ..... Table 8.5-3
Gamma Emitters ..... Table 8.5-3
Iron-55 ..... Table 8.5-3
Lead-210 ..... Table 8.5-3
Neptunium-237 ..... Table 8.5-3
Promethium-147 ..... Table 8.5-3
Plutonium-238, 239/240 ..... Table 8.5-3
Radium-226 ..... Table 8.5-3
Radium-228 ..... Table 8.5-3
Strontium-89, 90 and Total Strontium ..... Table 8.5-3
Technetium-99 ..... Table 8.5-3
Thorium-227, 228, 230, 232 ..... Table 8.5-3
Total Uranium ..... Table 8.5-3
Tritium ..... Table 8.5-3
Uranium-233/234, 235/236 ..... Table 8.5-3
Uranium-238 ..... Table 8.5-3

TCLP and SPLP

Mercury ..... Table 8.5-5
Metals (except mercury) ..... Table 8.5-5
Semivolatiles ..... Table 8.5-5
Volatiles ..... Table 8.5-5

Index (continued)

Page/Location

Samples
- Containers (Sample) 55
- Containers (Shipping) 57
- Disposal 63
- Field Collection 54
- Handling 58
- Holding Times (Also see Sample Containers, Preservatives, and Holding Times) 57-58
- Interlaboratory Transfers 62-63
- Log-In 59
- Preservatives (Also see Sample Containers, Preservatives, and Holding Times) 57
- Quality Control Samples (See Quality Control Samples)
- Radioactive 55
- Receipt 58-59
- Storage 59, 62

Software (Also see Computer)
- Changes (Control of) 32
- Documentation 33
- Industry Standard Software 32
- Quanterra® -Developed Software 32
- Revalidation 32
- Security 31
- Use 31
- Validation 31
- Verification 31
- Viruses 33

Standard Operating Procedures 13, 43

Standard Reporting Limit (See Reporting Limit)

Index (continued)

Page/Location

Standards
- Chemical Reference Standards 64
- Physical Reference Standards 64
- Reference Materials 22-23, 64
- Standard Verification 65

Statement of Management Position on Quality 1

Subcontractor (See Procurement)

Training
- Associate 15
- Files 17
- Health and Safety 17
- Individual Training Records 17-18
- Laboratory Staff 15
- Orientation 15
- QA Manager 17
- Qualification, Associate 15
- Quality 16
- Quality Orientation 16
- Records 18

Vision Statement 1

Vendor 19
- Partnerships 24
- Selection of 19

Waste Disposal 77

Water (See Internal QC Requirements)

Glossary

Glossary (continued)

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Glossary (continued)

acceptance limits

Data quality limits specified for analytical method performance.

accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

aliquot, aliquant

A measured portion of a sample taken for analysis.

analytical spike

A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. (Also see matrix spike.)

anomaly

See nonconformance.

areas needing improvement

Represent isolated instances of noncompliance or issues that are judged to have a less immediate impact on data quality. Laboratory management must correct the situation or otherwise ensure that the condition does not recur. This term replaces the previous term used "Observations."

arithmetic mean

The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

where: $\bar{x}$ = the mean

x_i = the ith data value

n = number of data values

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

Glossary (continued)

assessment
The evaluation process used to measure the performance or effectiveness of a system and its elements. Assessment is used as an all-inclusive term to denote any of the following: performance, systems, data and compliance audits, management systems reviews, peer reviews, inspections, or spot assessments.

associate
Employee.

audit
A planned and documented investigative evaluation of an item or process to determine its adequacy and effectiveness as well as compliance with established procedures, instructions, drawings, quality management plans, and other applicable documents.

benchmarking
A step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices.

bias
A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.

blind performance evaluation sample
A sample either submitted to the laboratory or prepared in the laboratory whereby the concentrations of parameters of concern are known by the preparer and not by the laboratory.

calibration
Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels at which actual measurements are to be made. Calibration is also the act of making a scheduled comparison of instrument performance against national standards for instruments

Glossary (continued)

which measure physical parameters such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

calibration curve
The graphical relationship between the known values for a series of calibration standards and instrument responses.

calibration factor (CF)
The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations.

$$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$

calibration standard
A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to standard reference materials (provided by NIST, or other recognized standards agencies) or a primary standard.

Certificate of Analysis
The standard Quanterra® format for reporting analytical results.

certified reference material
A reference material accompanied by a certificate issued by an organization certifying the contents and concentration(s) of the material. (See also standard reference material.)

chain-of-custody (COC)
A system of documentation demonstrating the physical custody and traceability of samples.

Glossary (continued)

check standard analyses
A standard (often a midpoint standard) analyzed at a frequency specified in the method or in a SOP to verify the continuing calibration of the standard curve.

client
Any individual or organization for whom items or services are furnished or work is performed in response to defined requirements and expectations.

client sample
The material or collection media submitted to the laboratory for analysis. Field QC samples are considered client samples but laboratory QC samples are not counted as client samples when counting samples for QC batches.

coefficient of variation (relative standard deviation)
A measure of precision (relative dispersion). It is equal to the standard deviation (s) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.

$$CV\,(RSD) = \left(\frac{s}{\bar{x}}\right) x\ 100$$

collocated samples
Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

comparability
Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

Glossary (continued)

completeness

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed. It is usually expressed as a percentage:

$$\%\ Completeness = \frac{V}{n} x\ 100$$

where: V = number of measurements judged valid
n = total number of measurements

composite

A sample composed of two or more increments.

control chart

A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.

control table

A tabular presentation of test results with respect to time or sequence of measurement, together with limits within which the results are expected to lie when the analytical process is in a state of control.

controlled document

A document for which the distribution is known. Updates of the document are sent to the original recipients, unless the copy distributed is an uncontrolled copy.

corrective action

A measure taken to rectify conditions adverse to quality and, where necessary, to preclude their recurrence.

Glossary (continued)

correlation coefficient
The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.

customer
See client.

data quality objective (DQO)
Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994). Typically, DQOs are identified during project scope and development of sampling and analysis plans. In this QA manual, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. Quanterra incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in Section 8.4 and are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield tracers.

Index (continued)

Page/Location

Maintenance (continued)

Cold Vapor Atomic Absorption (CVAA) - Leeman PS 200 ..... Table 8.11-9
Cold Vapor Atomic Absorption (CVAA) - PE 5000 ..... Table 8.11-10
Conductance Meter ..... Table 8.11-21
Digestion Block ..... Table 8.11-29
Dissolved Oxygen Meter ..... Table 8.11-20
Flame Atomic Absorption (Flame AA) ..... Table 8.11-5
Flash Point Tester ..... Table 8.11-30
Fourier Transform Infrared Spectrometry (FTIR) ..... Table 8.11-25
Gas Chromatograph (GC) ..... Table 8.11-11
Graphite Furnace Atomic Absorption (GFAA) ..... Table 8.11-8
High Pressure Liquid Chromatography (HPLC) ..... Table 8.11-4
ICAP/MS ..... Table 8.11-6
ICP ..... Table 8.11-7
Ion Chromatograph (IC) ..... Table 8.11-1
LACHAT Auto Analyzer ..... Table 8.11-2
Mass Spectrometer (MS) ..... Table 8.11-12
Ovens ..... Table 8.11-17
pH Meter ..... Table 8.11-24
Radiological Analysis Equipment ..... Table 8.11-26
Refrigerator ..... Table 8.11-16
Sonicator ..... Table 8.11-14
Specific Digital Ion Analyzer ..... Table 8.11-18
Spectrophotometer ..... Table 8.11-23
Total Organic Carbon (TOC) Analyzer (OI 7000) ..... Table 8.11-27
Total Organic Halide (TOX) Analyzer ..... Table 8.11-3
TRAACS 800 Auto Analyzer ..... Table 8.11-13
Turbidimeter ..... Table 8.11-19
Walk-in Cooler ..... Table 8.11-16

Service Agreements 74

Method

Analytical ..... 43, Table 8.2-1
Modifications 44

Index (continued)

Page/Location

Method Detection Limit (See Reporting Limit)

Method of Standard Additions 53

Nonconformance 79
- Anomaly 79
- Condition Upon Receipt Report (CUR) ..... 59, Figure 8.5-2
- Corrective Action 79
- Deficiency 79
- Nonconformance Log 80
- Nonconformance Memo 80
- Responsibilities 80

Organization
- Chart (Also see Section 1 of the Facility-Specific Appendix C and Appendix A) ..... Figure 1.3-1
- Structure 2

Orientation (See Training)

Performance Evaluation Samples (Also See Section 6 of the Facility-Specific Appendix C) 81

Practical Quantitation Limit (See Reporting Limit)

Precision and Accuracy Measurements
- Accuracy ..... 45, Table 8.6-1
- Arithmetic Mean ..... Table 8.6-1
- Laboratory QC Measurements ..... Table 8.4-3
- Lower Control Limit (LCL) ..... Table 8.6-1
- Lower Warning Limit (LWL) ..... Table 8.6-1
- Matrix QC Measurements ..... Table 8.4-4
- Precision ..... 45, Table 8.6-1
- Standard Deviation ..... Table 8.6-1
- Upper Warning Limit (UWL) ..... Table 8.6-1
- Upper Control Limit (UCL) ..... Table 8.6-1

Index (continued)

Page/Location

Preservatives (See Sample Containers, Preservatives, and Holding Times)

Preventive Maintenance (See Maintenance)

Project Planning 35

Procedures (Standardization of) 87

Procurement (Also see Vendor) 19
- Procedures 21
- Reference Materials (See Standards)
- Role of Corporate Director of Contracts 21-21
- Role of Quanterra® Purchasing 21
- Selection of Vendors 19-20
- Subcontract Laboratory Service 23

Project Planning 35

Project-Specific Reporting Limit (See Reporting Limit)

Qualification, Associate (See Training)

Quality Assurance 11-12

Quality Assurance Management Plan (QAMP) 13

Quality Assurance Program or Project Plan (QAPP) 13-14

Quality Assurance Summary (QAS) 14

Quality Control Samples 48
- Contract Laboratory Program (CLP) QC Samples
  - Cyanide, Total ..... Table 8.4-7
  - GFAA ..... Table 8.4-7
  - ICAP ..... Table 8.4-7

Index (continued)

Page/Location

Quality Control Samples (continued)

Mercury (CVAA) ..... Table 8.4-7
PCDD/PCDF ..... Table 8.4-7
Pesticides/PCBs ..... Table 8.4-7
Semivolatiles by GC/MS ..... Table 8.4-7
Volatiles by GC/MS ..... Table 8.4-7

Field ..... 48, Table 8.4-1

Collocated Sample ..... 49, Table 8.4-1
Field Blank ..... 49, Table 8.4-1
Field Duplicate ..... 49, Table 8.4-1
Field Matrix Spike ..... 49, Table 8.4-1
Rinsate Blank ..... 49, Table 8.4-1
Split Sample ..... 50, Table 8.4-1
Trip Blank ..... 49, Table 8.4-1

Inorganic (QC Samples)

Acidity ..... Table 8.4-5
Alkalinity ..... Table 8.4-5
Ammonia ..... Table 8.4-5
Ammonia (TKN) ..... Table 8.4-5
Biochemical Oxygen Demand (BOD) ..... Table 8.4-5
Bromide ..... Table 8.4-5
Chemical Oxygen Demand (COD) ..... Table 8.4-5
Chloride ..... Table 8.4-5
Chlorine, Residual ..... Table 8.4-5
Chromium (Hexavalent) Cr^{+6} ..... Table 8.4-5
Color ..... Table 8.4-5
Conductivity ..... Table 8.4-5
Cyanide (Amenable) ..... Table 8.4-5
Cyanide (Total) ..... Table 8.4-5
Flashpoint ..... Table 8.4-5
Fluoride ..... Table 8.4-5
Hardness ..... Table 8.4-5
Iodide ..... Table 8.4-5
Iron, Phenanthroline Method ..... Table 8.4-5
Methylene Blue Active Substances (MBAS) ..... Table 8.4-5

Index (continued)

Page/Location

Quality Control Samples (continued)

Metals ..... Table 8.4-5
Flame AA ..... Table 8.4-5
Graphite Furnace AA ..... Table 8.4-5
ICP ..... Table 8.4-5
ICP/MS ..... Table 8.4-5
Mercury (CVAA) ..... Table 8.4-5
Nitrate ..... Table 8.4-5
Nitrite ..... Table 8.4-5
Nitrate-Nitrite ..... Table 8.4-5
Odor ..... Table 8.4-5
pH ..... Table 8.4-5
Phenolics ..... Table 8.4-5
Phosphate ..... Table 8.4-5
Phosphorous (Total and Orthophosphate) ..... Table 8.4-5
Reactivity (Cyanide and Sulfide) ..... Table 8.4-5
Silica, Dissolved ..... Table 8.4-5
Solids ..... Table 8.4-5
Specific Conductance ..... Table 8.4-5
Sulfate ..... Table 8.4-5
Sulfide ..... Table 8.4-5
Sulfite ..... Table 8.4-5
Temperature ..... Table 8.4-5
Total Organic Carbon (TOC) ..... Table 8.4-5
Total Organic Halides (TOX) ..... Table 8.4-5
Turbidity ..... Table 8.4-5
Water Content ..... Table 8.4-5

Laboratory QC Samples ..... 50, Table 8.4-2, Table 8.4-3
Analytical Spike ..... 53, Table 8.4-2
Duplicate ..... 53, Table 8.4-2, Table 8.4-4
Instrument/Calibration Blank ..... 51, Table 8.4-2, Table 8.4-3
Interference Check Sample ..... 53, Table 8.4-2
Internal Standard ..... 53, Table 8.4-2
Laboratory Control Sample ..... 52, Table 8.4-2, Table 8.4-3
Laboratory Performance QC Samples ..... Table 8.4-3

Index (continued)

Page/Location

Quality Control Samples (continued)
Matrix Spike ..... 52, Table 8.4-2, Table 8.4-4
Matrix Spike Duplicate ..... 52, Table 8.4-2, Table 8.4-4
Method Blank ..... 51, Table 8.4-2, Table 8.4-3
Method of Standard Additions 53
Post Digestion Spike (See Analytical Spike)
Surrogate ..... 53, Table 8.4-2
Matrix QC Samples ..... Table 8.4-4
Organic QC Samples
Aromatic Volatiles by GC ..... Table 8.4-6
Dioxins/Dibenzofurans by HRGC/HRMS ..... Table 8.4-6
Dioxins/Dibenzofurans by HRGC/LRMS ..... Table 8.4-6
Halogenated Volatiles by GC ..... Table 8.4-6
Herbicides ..... Table 8.4-6
Nitroaromatics by HPLC ..... Table 8.4-6
Organophosphorous Pesticides ..... Table 8.4-6
Polyaromatic Hydrocarbons (PAHs) by HPLC and GC ..... Table 8.4-6
Pesticides/PCBs ..... Table 8.4-6
Petroleum Hydrocarbons ..... Table 8.4-6
Purgeable Halocarbons by GC ..... Table 8.4-6
Acrolein and Acrylonitrile ..... Table 8.4-6
Semivolatiles ..... Table 8.4-6
Volatiles by GC/MS ..... Table 8.4-6
Radiological QC Samples
(Also See Section 7 of the Facility-Specific Appendix C, where applicable) 54
Carrier 54
Tracer 54
Yield Monitors ..... 54, Table 8.4-2

Quality Document (See Document, Quality)

Quality Improvement 87

Quality Management System (QMS) 1

Index (continued)

Page/Location

Quality Organization 2

Quality Policy Document 11

Quality-Related Item (QRI) ..... 20, Table 4.2-1

Quality Report to Management 84

Radioisotope ..... 54, Table 8.5-3

Record
- Attendance (See Training, Attendance Records)
- Disposal 29
- Project 28
- Management 27
- Quality 27
- Retention 29

Reference Material (See Standards)

Reference Standards (See Standards)

Reporting Limit 47
- Maximum Contaminant Level (MCL) 47
- Method Detection Limit (MDL) 46
- Minimum Detectable Concentration 48
- Project-Specific Reporting Limit (PSRL) 47
- Radiochemical 47-48

Resume (Associate) 17

Index (continued)

Page/Location

Sample Containers, Preservations, and Holding Times

Contract Laboratory Program (CLP)

Cyanide, Total and Amenable to Chlorination ..... Table 8.5-4
PCDD/PCDF ..... Table 8.5-4
GFAA ..... Table 8.5-4
ICAP ..... Table 8.5-4
Mercury (CVAA) ..... Table 8.5-4
Pesticides/PCBs ..... Table 8.5-4
Semivolatiles ..... Table 8.5-4
Volatiles ..... Table 8.5-4

Inorganic (Sample Containers, Preservations, and Holding Times)

Acidity ..... Table 8.5-1
Alkalinity ..... Table 8.5-1
Ammonia ..... Table 8.5-1
Biochemical Oxygen Demand (BOD) ..... Table 8.5-1
Bromide ..... Table 8.5-1
Chemical Oxygen Demand (COD) ..... Table 8.5-1
Chloride ..... Table 8.5-1
Chlorine, Residual ..... Table 8.5-1
Chromium (Hexavalent) Cr^{+6} ..... Table 8.5-1
Color ..... Table 8.5-1
Conductivity ..... Table 8.5-1
Cyanide (Amenable) ..... Table 8.5-1
Cyanide (Total) ..... Table 8.5-1
Flashpoint (Ignitability) ..... Table 8.5-1
Fluoride ..... Table 8.5-1
Hardness (Total) ..... Table 8.5-1
Iodide ..... Table 8.5-1
Iron, Phenanthroline Method ..... Table 8.5-1
Methylene Blue Active Substances (MBAS) ..... Table 8.5-1
Metals ..... Table 8.5-1
Mercury (CVAA) ..... Table 8.5-1
Nitrate ..... Table 8.5-1
Nitrite ..... Table 8.5-1

Index (continued)

Page/Location

Sample Containers, Preservations, and Holding Times (continued)

Nitrate-Nitrite ..... Table 8.5-1
Odor ..... Table 8.5-1
Orthophosphate ..... Table 8.5-1
pH ..... Table 8.5-1
Phenolics ..... Table 8.5-1
Phosphate ..... Table 8.5-1
Phosphorous (Total) ..... Table 8.5-1
Reactivity (Cyanide and Sulfide) ..... Table 8.5-1
Settleable Solids ..... Table 8.5-1
Silica, Dissolved ..... Table 8.5-1
Specific Conductance ..... Table 8.5-1
Sulfate ..... Table 8.5-1
Sulfide ..... Table 8.5-1
Sulfite ..... Table 8.5-1
Temperature ..... Table 8.5-1
Total Dissolved Solids ..... Table 8.5-1
Total Kjeldahl Nitrogen (TKN) ..... Table 8.5-1
Total Organic Carbon (TOC) ..... Table 8.5-1
Total Organic Halides (TOX) ..... Table 8.5-1
Total Solids ..... Table 8.5-1
Total Suspended Solids ..... Table 8.5-1
Total Volatile Solids ..... Table 8.5-1
Turbidity ..... Table 8.5-1
Water Content ..... Table 8.5-1

Organic

Aromatic Volatiles ..... Table 8.5-2
Dioxins/Dibenzofurans ..... Table 8.5-2
Halogenated Volatiles ..... Table 8.5-2
Herbicides ..... Table 8.5-2
Nitroaromatics ..... Table 8.5-2
Organophosphorous Pesticides ..... Table 8.5-2
Pesticides/PCBs ..... Table 8.5-2
Petroleum Hydrocarbons/Oil and Grease ..... Table 8.5-2

Index (continued)

Page/Location

Sample Containers, Preservations, and Holding Times (continued)

Polyaromatic Hydrocarbons (PAH) by GC and HPLC ..... Table 8.5-2
Purgeable Halocarbons ..... Table 8.5-2
Acrolein and Acrylonitrile ..... Table 8.5-2
Semivolatiles ..... Table 8.5-2
Volatiles ..... Table 8.5-2

Radiological

Alpha/Beta (Gross) ..... Table 8.5-3
Americium-241 ..... Table 8.5-3
Carbon-14 ..... Table 8.5-3
Calcium-45 ..... Table 8.5-3
Curium-242 ..... Table 8.5-3
Gamma Emitters ..... Table 8.5-3
Iron-55 ..... Table 8.5-3
Lead-210 ..... Table 8.5-3
Neptunium-237 ..... Table 8.5-3
Promethium-147 ..... Table 8.5-3
Plutonium-238, 239/240 ..... Table 8.5-3
Radium-226 ..... Table 8.5-3
Radium-228 ..... Table 8.5-3
Strontium-89, 90 and Total Strontium ..... Table 8.5-3
Technetium-99 ..... Table 8.5-3
Thorium-227, 228, 230, 232 ..... Table 8.5-3
Total Uranium ..... Table 8.5-3
Tritium ..... Table 8.5-3
Uranium-233/234, 235/236 ..... Table 8.5-3
Uranium-238 ..... Table 8.5-3

TCLP and SPLP

Mercury ..... Table 8.5-5
Metals (except mercury) ..... Table 8.5-5
Semivolatiles ..... Table 8.5-5
Volatiles ..... Table 8.5-5

Index (continued)

Page/Location

Samples
- Containers (Sample) 55
- Containers (Shipping) 57
- Disposal 63
- Field Collection 54
- Handling 58
- Holding Times (Also see Sample Containers, Preservatives, and Holding Times) 57-58
- Interlaboratory Transfers 62-63
- Log-In 59
- Preservatives (Also see Sample Containers, Preservatives, and Holding Times) 57
- Quality Control Samples (See Quality Control Samples)
- Radioactive 55
- Receipt 58-59
- Storage 59, 62

Software (Also see Computer)
- Changes (Control of) 32
- Documentation 33
- Industry Standard Software 32
- Quanterra® -Developed Software 32
- Revalidation 32
- Security 31
- Use 31
- Validation 31
- Verification 31
- Viruses 33

Standard Operating Procedures 13, 43

Standard Reporting Limit (See Reporting Limit)

Index (continued)

Page/Location

Standards
- Chemical Reference Standards 64
- Physical Reference Standards 64
- Reference Materials 22-23, 64
- Standard Verification 65

Statement of Management Position on Quality 1

Subcontractor (See Procurement)

Training
- Associate 15
- Files 17
- Health and Safety 17
- Individual Training Records 17-18
- Laboratory Staff 15
- Orientation 15
- QA Manager 17
- Qualification, Associate 15
- Quality 16
- Quality Orientation 16
- Records 18

Vision Statement 1

Vendor 19
- Partnerships 24
- Selection of 19

Waste Disposal 77

Water (See Internal QC Requirements)

Glossary

Glossary (continued)

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Glossary (continued)

acceptance limits

Data quality limits specified for analytical method performance.

accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

aliquot, aliquant

A measured portion of a sample taken for analysis.

analytical spike

A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. (Also see matrix spike.)

anomaly

See nonconformance.

areas needing improvement

Represent isolated instances of noncompliance or issues that are judged to have a less immediate impact on data quality. Laboratory management must correct the situation or otherwise ensure that the condition does not recur. This term replaces the previous term used "Observations."

arithmetic mean

The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

where: $\bar{x}$ = the mean

x_i = the ith data value

n = number of data values

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

Glossary (continued)

assessment
The evaluation process used to measure the performance or effectiveness of a system and its elements. Assessment is used as an all-inclusive term to denote any of the following: performance, systems, data and compliance audits, management systems reviews, peer reviews, inspections, or spot assessments.

associate
Employee.

audit
A planned and documented investigative evaluation of an item or process to determine its adequacy and effectiveness as well as compliance with established procedures, instructions, drawings, quality management plans, and other applicable documents.

benchmarking
A step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices.

bias
A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.

blind performance evaluation sample
A sample either submitted to the laboratory or prepared in the laboratory whereby the concentrations of parameters of concern are known by the preparer and not by the laboratory.

calibration
Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels at which actual measurements are to be made. Calibration is also the act of making a scheduled comparison of instrument performance against national standards for instruments

Glossary (continued)

which measure physical parameters such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

calibration curve
The graphical relationship between the known values for a series of calibration standards and instrument responses.

calibration factor (CF)
The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations.

$$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$

calibration standard
A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to standard reference materials (provided by NIST, or other recognized standards agencies) or a primary standard.

Certificate of Analysis
The standard Quanterra® format for reporting analytical results.

certified reference material
A reference material accompanied by a certificate issued by an organization certifying the contents and concentration(s) of the material. (See also standard reference material.)

chain-of-custody (COC)
A system of documentation demonstrating the physical custody and traceability of samples.

Glossary (continued)

check standard analyses
A standard (often a midpoint standard) analyzed at a frequency specified in the method or in a SOP to verify the continuing calibration of the standard curve.

client
Any individual or organization for whom items or services are furnished or work is performed in response to defined requirements and expectations.

client sample
The material or collection media submitted to the laboratory for analysis. Field QC samples are considered client samples but laboratory QC samples are not counted as client samples when counting samples for QC batches.

coefficient of variation (relative standard deviation)
A measure of precision (relative dispersion). It is equal to the standard deviation (s) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.

$$CV\,(RSD) = \left(\frac{s}{\bar{x}}\right) x\ 100$$

collocated samples
Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

comparability
Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

Glossary (continued)

completeness

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed. It is usually expressed as a percentage:

$$\%\,Completeness = \frac{V}{n} x\ 100$$

where: V = number of measurements judged valid
n = total number of measurements

composite

A sample composed of two or more increments.

control chart

A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.

control table

A tabular presentation of test results with respect to time or sequence of measurement, together with limits within which the results are expected to lie when the analytical process is in a state of control.

controlled document

A document for which the distribution is known. Updates of the document are sent to the original recipients, unless the copy distributed is an uncontrolled copy.

corrective action

A measure taken to rectify conditions adverse to quality and, where necessary, to preclude their recurrence.

Glossary (continued)

correlation coefficient
The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.

customer
See client.

data quality objective (DQO)
Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994). Typically, DQOs are identified during project scope and development of sampling and analysis plans. In this QA manual, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. Quanterra incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in Section 8.4 and are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield tracers.

Glossary (continued)

data validation
See validation - data.

data verification
See verification - data.

deficiency
See nonconformance or finding.

degrees of freedom
The number of independent deviations used in calculating an estimate of the standard deviation.

double blind performance evaluation sample
A sample that contains select parameters at defined levels. The levels are unknown to the laboratory. The laboratory is also unaware that the sample is a performance evaluation sample.

duplicate sample analyses
Different aliquots of the same sample are analyzed to evaluate the precision of an analysis.

error
The difference between an observed or measured value and its true value.

field blank
A blank that is prepared and handled in the field and analyzed in the same manner as its corresponding client samples.

field matrix spike
A sample created by spiking target analytes into a sample in the field at the point of sample acquisition.

Glossary (continued)

finding
Noncompliant practices or policies which have significant adverse impact on data quality, technical defensibility, or regulatory acceptance of data. Findings require immediate attention by the laboratory management and must be resolved to comply with Quanterra®'s quality documents and laboratory-established procedures often called deficiencies by auditors.

geometric mean
The nth root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much population data.

initial calibration
Analysis of a series of analytical standards at different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds prior to the analysis of samples.

inspection
Examination or measurement of an item or activity to verify conformance to specific requirements.

instrument detection limit (IDL)
The smallest concentration or amount an instrument can reliably detect.

internal standard (IS)
A compound added to every standard, QC sample, client sample, or sample extract at a known concentration prior to analysis for the purpose of quantitation. For example, internal standards are used as the basis for quantitation of the target compounds by GC/MS.

linear regression
A statistical method for finding a straight line that best fits a set of two or more data points, thus providing a relationship between two or more variables.

Glossary (continued)

matrix
The component or substrate which contains the analyte(s) of interest. Examples of matrices are water, soil or sediment, and air. Matrix is not synonymous with phase (liquid or solid).

matrix effect
An interference in the measurement of analyte(s) in a sample that is caused by materials in the sample. Matrix effects may cause elevated reporting limits or may prevent the acquisition of acceptable results.

matrix spike (MS)
An aliquot of a matrix fortified (spiked) with known quantities of specific compounds and subjected to an entire analytical procedure in order to indicate the appropriateness of the method for a particular matrix. The percent recovery for the respective compound(s) is then calculated.

matrix spike duplicate (MSD)
A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

may
Denotes permission but not a requirement.

mean
See arithmetic mean.

measurement
The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

median
The middle value of a set of data when the data set is ranked in increasing or decreasing order.

Glossary (continued)

method
An assemblage of techniques.

method blank (MB)
An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. Examples of method blanks are a volume of deionized or distilled laboratory water for water samples, a purified solid matrix for soil/sediment samples, or a generated zero air.

method detection limit (MDL)
The minimum concentration of an analyte that, in a given matrix and with a specific method, can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is operationally defined as:

$$MDL = st_{(n-1,\ \alpha=0.99)}$$

where:

s = the standard deviation of a number of measurements of a blind or sample matrix containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1,\ \alpha=0.99)}$ = the student's value for a one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99% confidence level and n-1 degrees of freedom.

modified method
A standard or reference method which has been changed to meet project or matrix requirements.

must
Denotes a requirement is mandatory and has to be met.

Glossary (continued)

notable practices

Laboratory practices that increase effectiveness and quality and represent improvements with respect to conventional laboratory operations.

nonconformance

An unplanned deviation from an established protocol or plan. The deviation may be the result of Quanterra's® actions, then termed a deficiency. If the deviation is the result of events beyond the control of Quanterra®, it is termed an anomaly.

operational calibration

Routinely performed as part of instrument usage, such as the development of a standard calibration curve. Operational calibration is generally performed for instrument systems.

outlier

A result excluded from the statistical calculations due to being deemed "suspicious" when applying the "Grubbs Test" (or equivalent).

parameter

A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

percent difference

When two independent measurements of the same characteristics are available, it is possible to use the percent difference instead of the coefficient of variation to measure precision.

$$\%D = \left| \frac{X_1 - X_2}{X_1} \right| x\ 100\%$$

where: $\%D$ = percent difference

X_1 = first value

X_2 = second value

Glossary (continued)

percent recovery
A measure of accuracy determined from the comparison of a reported spike value to its true spike concentration.

$$\%R = \frac{observed\ conc. - sample\ conc.}{true\ spike\ conc.} \times 100\%$$

performance audit
See performance evaluation.

performance evaluation (PE)
A type of audit in which a known or characterized value is compared to the result obtained through the routine analysis of the sample in the laboratory to evaluate the proficiency of an analyst or laboratory.

periodic calibration
A calibration that is performed at prescribed intervals for equipment such as balances, thermometers, and balance weights. In general, they are performed on equipment that are distinct, singular purpose units, and are relatively stable in performance.

population
A generic term denoting any finite or infinite collection of individual things, objects, or events.

practical quantitation limit (PQL)
The lowest concentration a method can reliably achieve within limits of precision and accuracy and is derived from empirical, matrix-free method performance studies.

precision
Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent

Glossary (continued)

difference (RPD) for duplicate measurements. Table 8.6-1 illustrates the formulae used to calculate units of precision (i.e., RSD and RPD).

preventive maintenance
An organized program within Quanterra® laboratories of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use.

primary standard
A material having a known, stable property that can be accurately measured or derived from established physical or chemical constants. It is readily reproducible and can be accepted (within stated limits) and used to establish the same value of another substance or item.

procedure
Detailed instructions to permit replication of a method. (See standard operating procedure.)

proficiency testing
A series of planned tests which will determine the ability of field technicians or laboratory analysts to perform routine analyses. The results from this testing may be used for comparison against established criteria or for relative comparisons among the data from a group of technicians or analysts.

project-specific reporting limit (PSRL)
See reporting limit.

protocol
Methodology specified in regulatory, authoritative, or contractual situations.

QC batch
The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period.

Glossary (continued)

QC check sample
A reference matrix containing known concentrations of parameters of interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria, corrective actions are taken.

qualification (personnel)
The characteristics of abilities gained through education, training, or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

quality
The sum of features and properties/characteristics of a process, item, or service that bears on its ability to meet the stated needs of the user. Quanterra® has defined quality as meeting the needs of our clients, both internal and external.

quality assurance (QA)
An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

Quality Assurance Directive
QA directives are memos issued by the QA Director (or the QA Managers for their facility) to clarify policies, Procedures, and the QAMP; or to give direction for an immediate action to ensure or maintain quality.

Quality Assurance Management Plan (QAMP)
The Quality Assurance Management Plan for Environmental Analyses (QAMP) is a formal document that describes quality systems in terms of organizational structure, functional responsibilities of management, and staff, and lines of authority. The QAMP documents the QMS and describes both the organizational and project-specific principles, goals, controls, and tools of the QMS. The QAMP provides the criteria and specifications for the generation of environmental analytical data.

Glossary (continued)

Quality Assurance Project or Program Plan (QAPP)
A formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure the results of the work performed will satisfy the stated performance criteria.

quality control (QC)
The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that it meets the stated requirements established by the client or by Quanterra®.

quality improvement
The process of improving the quality of operations. This process encourages worker recommendations for improvement of work processes and requires timely management evaluation and feedback or implementation.

quality management
That aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality management system.

quality management system (QMS)
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

random error
Variations of repeated measurements that are random in nature and individually not predictable.

Glossary (continued)

range
The difference between the largest and smallest numbers in a set of numbers.

raw data
All documentation associated with the original recording of analytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

reagent water
Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. The reagent water's purity and acceptability is verified by analysis with each set of samples.

recovery
See percent recovery.

reference method
A method of known and demonstrated accuracy.

regression coefficients
The quantities describing the slope and intercept of a regression line.

relative error
An error expressed as a percentage of the true value or accepted reference value.

relative percent different (RPD)
Statistic for evaluating the precision of a replicate set. For replicate results:

$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$

Glossary (continued)

where: X_1 = first observed concentration
X_2 = second observed concentration

relative response factor (RRF)
A measure of the relative mass spectral response of a compound compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. Because a RRF is the comparison of two responses, it is a unitless number. RRFs are determined by the following equation:

$$RRF = \frac{A_x}{A_{IS}} x \frac{C_{IS}}{C_x}$$

where: A = area of the characteristic ion measured
C = concentration
IS = internal standard
x = analyte of interest

relative standard deviation (RSD)
See coefficient of variation.

reporting limit (RL)
One of two types of reporting limit conventions within Quanterra®. The Reporting Limit (RL) is a uniform, Quanterra® -wide reporting limit based on an evaluation of the PQLs at Quanterra® laboratories and the expected method performance in routine water and soil matrices. Project Specific Reporting Limits (PSRLs) are reporting limits that are defined by project requirements.

representative sample
A sample taken to represent a lot or population as accurately and precisely as possible.

Glossary (continued)

representativeness
Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At each Quanterra laboratory, every effort must be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

reproducibility
The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

response factor (RF)
A factor derived from the calibration of a compound that is used in the quantitation calculation of sample analytes. A response factor may be derived from an external standard calibration (then called a Calibration Factor) or from an internal standard calibration (then called a Relative Response Factor).

secondary standard
A material having a property that is calibrated against a primary standard.

self assessment
Assessments of work conducted by individuals, groups, or organizations directly responsible for overseeing or performing the work.

shall
Denotes a requirement that is mandatory and has to be met.

should
Denotes a guideline or recommendation.

Glossary (continued)

standard addition

The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish, by extrapolation of the plotted responses, the level of the analyte of interest present in the original sample.

standard deviation

A measure of the dispersion about the mean of the elements in a population. The square root of the variance of a set of values:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

where: s = standard deviation
Σ = sum of
X = observed values
n = number of observations

standardization

The establishment of the value of a potential standard with respect to an established or known standard.

standard method

A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.

standard operating procedure (SOP)

A written document that details an operation, analysis, or action, with prescribed techniques and steps, that is officially approved as the method for performing certain routine or repetitive tasks.

Glossary (continued)

standard reference material (SRM)
A material produced in quantity, of which certain properties have been certified by the National Institute of Standards and Technology (NIST), formerly NBS, or other agencies to the extent possible to satisfy its intended use.

standard verification
Standard is checked by Quanterra® or the vendor versus a known specification. See Section 8.5.4.3.

statistic
A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.

stock solution
A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.

subsample
A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

supplier
See vendor.

surrogate (surrogate standard)
Compounds, when required by a method, that are used added to every blank, sample, LCS, matrix spike, matrix spike duplicate, and standard. They are used to evaluate analytical efficiency by measuring recovery. Surrogates include brominated, fluorinated, or isotopically-labeled compounds that are not expected to be detected in environmental media.

systematic error
The condition of a consistent deviation of the results of a measurement process from the reference or known level.

Glossary (continued)

systems audit or evaluation
A systematic on-site qualitative review of facilities, procedures, equipment, training, record keeping, data verification, and reporting aspects of a quality assurance system to arrive at a measure of the capability of the system. Within Quanterra®, system audits or evaluations are performed on a periodic basis under the direction of the Quanterra® Corporate Director of Quality Assurance.

technique
Physical or chemical principle for characterizing materials of chemical systems.

traceability of data
The entire documented chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction, and storage activities. The documentation will allow complete reconstruction of the data.

traceability of samples
During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers. These sample numbers shall be accompanied by documentation (chain-of-custody form) which clearly identifies all parameters associated with sample acquisition. All additional sample numbering systems applied to the sample must be clearly cross-referenced to the field sample number to provide for traceability of samples from acquisition to reporting of sample results.

traceability of standards
The ability of an analytical standard material used for calibration purposes to be traced to its source. The standards used by Quanterra® must be traceable via written documentation to sources which produce or sell verified or certified standards, i.e., National Institute for Standards and Technology, or vendors preparing standards from those sources which they have certified.

validation - computer software
The process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality

Glossary (continued)

attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.

validation - data
The process of a second party performing a systematic review of the raw and final data produced by a laboratory using predetermined criteria to ascertain the validity of the data with respect to the criteria (e.g., HAZWRAP data validation).

vendor
Any individual or organization furnishing items or services or performing work according to a procurement document. This is an all-inclusive term used in place of any of the following: supplier, seller, contractor, subcontractor, or consultant.

verification - computer software
The process of checking the accuracy of manually entered or automatically (electronically) calculated information.

verification - data
The process of reviewing data to ensure that data reduction has been correctly performed and that analytical results to be reported correspond to the data acquired and processed.

Appendix A

Quanterra®
Quality Assurance Management Plan

Corporate Key Personnel List and Quanterra® Operations Organizational Chart

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Quanterra®
Corporate Key Personnel List

Associate Name	Title	Degree	Years of Experience
Gerhard F. König	President and CEO	MBA	33
Enos Tracey	Chief Financial Officer	BS, Accounting MBA, Accountancy	14
Elizabeth Counts	Human Resources Manager	MS, Human Resources	9
Mark Matthews	Chief Operating Officer	BA, Accounting	19
Chris M. Lee	Vice President Operations Services	Business/Biology	14
Donnie Heinrich	Senior Vice President of Sales	BS, Chemistry	23
Brad S. Figley	Senior Vice President Legal and Strategy	JD in Law	17
Bill Henning	Director of Information Technology	BA, Sociology MBA, Quantitative Studies	15
Jack Hall	Corporate Director of Quality Assurance	BS, Chemistry	34
Don Dihel	Corporate Director of Environmental Health and Safety	BA, Chemistry	24
Richard Burrows	Technology/Principal Scientist	BS, Chemistry Ph.D., Analytical Chemistry	13

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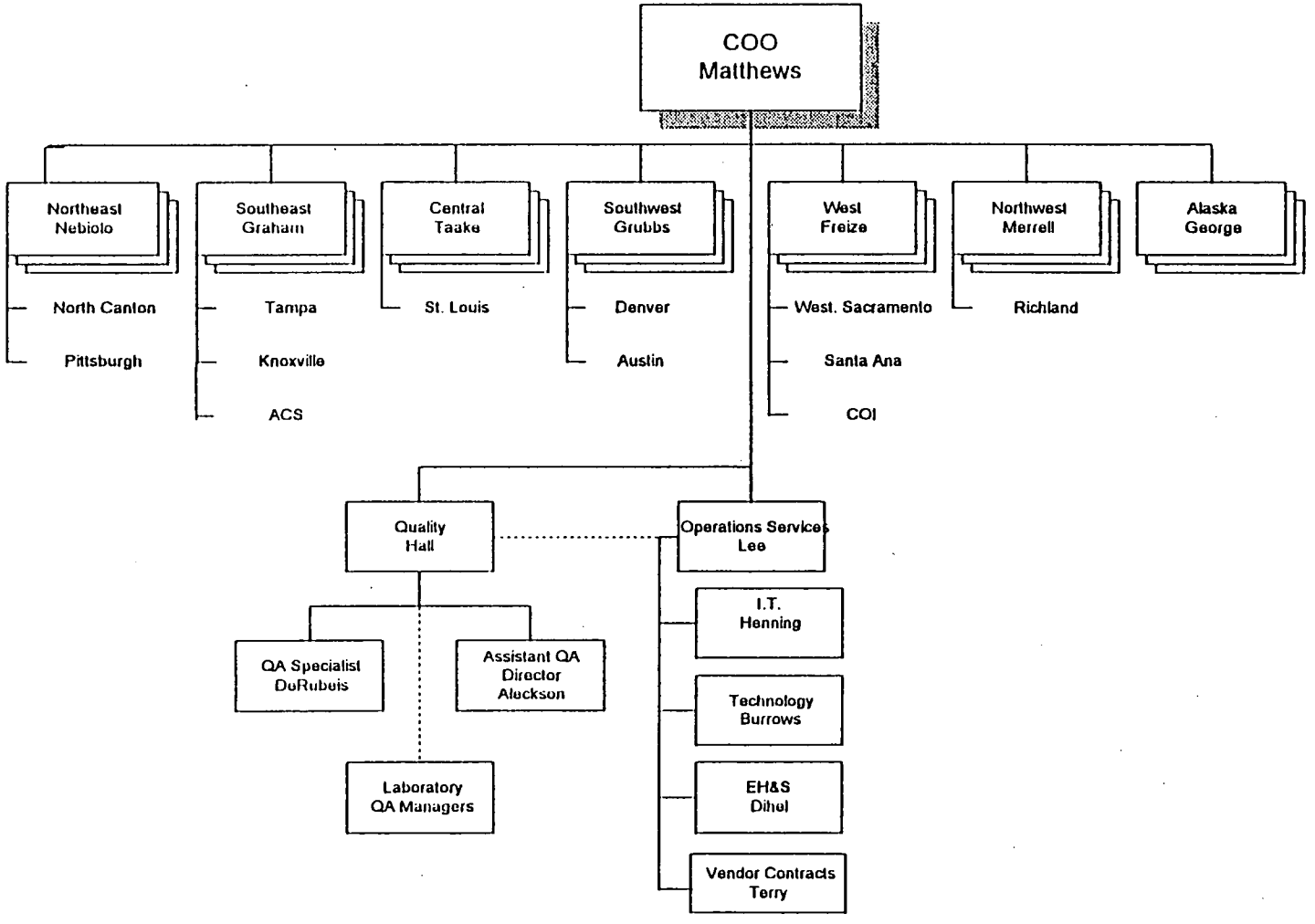
QUANTERRA OPERATIONS ORGANIZATIONAL CHART



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Appendix B

Quanterra®
Quality Assurance Management Plan

Addresses of Quanterra® Locations

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APPENDIX B

ADDRESSES OF Quanterra® LOCATIONS

LABORATORIES

Alaska
Quanterra®
5761 Silverado Way
Suite N
Anchorage, Alaska 99518
Voice: (907) 563-4800
Fax: (907) 563-4815

California
Quanterra®
18501 East Gale Avenue
Suite 130
City of Industry, California 91748
Voice: (818) 965-1006
Fax: (818) 965-1003

Quanterra®
1721 South Grand Avenue
Santa Ana, California 92705
Voice: (714) 258-8610
Fax: (714) 258-0921

Quanterra®
880 Riverside Parkway
West Sacramento, California 95605
Voice: (916) 373-5600
Fax: (916) 372-1059

Colorado
Quanterra®
4955 Yarrow Street
Arvada, Colorado 80002
Voice: (303) 421-6611
Fax: (303) 431-7171

Florida
Quanterra®
5910 H Breckenridge Parkway
Tampa, Florida 33610
Voice: (813) 621-0784
Fax: (813) 623-6021

Missouri
Quanterra®
13715 Rider Trail North
Earth City, Missouri 63045
Voice: (314) 298-8566
Fax: (314) 298-8757

Ohio
Quanterra®
4101 Shuffel Drive, N.W.
North Canton, Ohio 44720
Voice: (330) 497-9396
Fax: (330) 497-0772

Pennsylvania
Quanterra®
450 William Pitt Way, Building 6
Pittsburgh, Pennsylvania 15238
Voice: (412) 826-5477
Fax: (412) 826-5571

Philadelphia Service Center
301 Chelsea Park Way
Boothwyn, PA 19061
Voice: (610) 485-7312
Fax: (610) 485-7314

LABORATORIES (Continued)

Tennessee
Quanterra®
5815 Middlebrook Pike
Knoxville, Tennessee 37921
Voice: (423) 588-6401
Fax: (423) 584-4315

Texas
Quanterra®
5307 Industrial Oaks Boulevard
Suite 160
Austin, Texas 78735
Voice: (512) 892-6684
Fax: (512) 892-6652

Washington
Quanterra®
2800 George Washington Way
Richland, Washington 99352
Voice: (509) 375-3131
Fax: (509) 375-5590

ANALYTICAL CONSULTING SERVICES

Tennessee
Quanterra®
5815 Middlebrook Pike
Knoxville, Tennessee 37921
Voice: (423) 588-6401
Fax: (423) 212-3085

CORPORATE HEADQUARTERS

Quanterra®
One DTC
5251 DTC Parkway
Suite 415
Englewood, Colorado 80111
Voice: (303) 796-2222
Fax: (303) 796-2002

Appendix C

Quanterra®
Quality Assurance Management Plan

Facility-Specific Appendix

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List of Laboratory Locations

This appendix contains facility-specific, quality-related information and requirements for Quanterra® laboratories. These laboratories are located in the following cities:

- Anchorage, Alaska
- Austin, Texas
- City of Industry, California
- Denver, Colorado
- Knoxville, Tennessee
- North Canton, Ohio
- Pittsburgh, Pennsylvania
- Richland, Washington
- Sacramento, California
- Santa Ana, California
- St. Louis, Missouri
- Tampa, Florida

Each laboratory section in this appendix contains information specific to that laboratory only and contains the following basic outline:

Section	Contents
0	Table of Contents
1	Organizational Chart
2	Instrument List
3	Standard Operating Procedures List
4	Analytical Methods
5	MDLs, RLs, and CRDLs
6	Performance Evaluation Studies
7	Additional Operation-Specific Information

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Facility Appendix

Quality Assurance Management Plan
Quanterra® Incorporated

Sacramento Laboratory
880 Riverside Parkway
West Sacramento, California 95605
(916) 373-5600

Approved by:

Pamela A. Schemmer

Pam Schemmer
Quality Assurance Manager
Sacramento Laboratory

Eric Redman
Laboratory Manager/Technical Director
Sacramento Laboratory

Jack R. Hall
Corporate Director of Quality Assurance
Phone Number: (423) 588-6401

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Facility Appendix
Sacramento Laboratory

Section Sacramento-0

Table of Contents

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List of Sections

Section No.	Contents	Section - Page
Sacramento - 0	Table of Contents	0-1
Sacramento - 1	Organizational Chart	1-1
Sacramento - 2	Instrument List	2-1
Sacramento - 3	Standard Operating Procedures	3-1
Sacramento - 4	Analytical Methods	4-1
Sacramento - 5	Method Detection Limits and Reporting Limits	5-1
Sacramento - 6	Performance Evaluation Studies	6-1
Sacramento - 7	Additional Operation-Specific Information	7-1

List of Figures

Figure No.	Title	Section - Page
Sacramento -1-1	Organizational Chart	1-3
Sacramento -7-1	Floor Plan	7-3

List of Tables

Table No.	Title	Section - Page
Sacramento -1-1	Key Personnel List	1-4
Sacramento -2-1	Instrument List	2-3
Sacramento -3-1	Standard Operating Procedures List	3-3
Sacramento -4-1	Organic Preparation and Clean-up Methods	4-3
Sacramento -4-2	Organic Methods	4-10
Sacramento -4-3	Metals Preparation Methods	4-14
Sacramento -4-4	Metals Methods	4-16
Sacramento -4-5	Wet Chemistry Methods	4-34
Sacramento -5-1	GC Volatiles CA LUFT and BTEX by 8021B Method Detection Limits (MDL) and Reporting Limits (RL)	5-3
Sacramento -5-2	Method 8021B Method Detection Limits (MDL) and Reporting Limits (RL)	5-4
Sacramento -5-3	Method 504.1 Method Detection Limits (MDL) and Reporting Limits (RL)	5-5
Sacramento -5-4	GC Semi-Volatile Method 8015M Method Detection Limits (MDL) and Reporting Limits (RL)	5-6
Sacramento -5-5	Method 8081A Organochlorine Pesticides Method Detection Limits (MDL) and Reporting Limits (RL)	5-7
Sacramento -5-6	Method 8082 Aroclors Method Detection Limits (MDL) and Reporting Limits (RL)	5-8
Sacramento -5-7	Modified 8151A Chlorinated Herbicides Method Detection Limits (MDL) and Reporting Limits (RL)	5-9
Sacramento -5-8	Method 8310 Polynuclear Aromatic Hydrocarbons Method Detection Limits (MDL) and Reporting Limits (RL)	5-10
Sacramento -5-9	8330 Nitroaromatics and Nitramines Method Detection Limits (MDL) and Reporting Limits (RL)	5-11
Sacramento -5-10	Method 8260B Volatile Organics by GC/MS Method Detection Limits (MDL) and Reporting Limits (RL)	5-12
Sacramento-5-11	Method 8270C Semivolatile Organics by GC/MS Method Detection Limits (MDL) and Reporting Limits (RL)	5-14
Sacramento -5-12	CWM Chemical Degradates by GC/MS Method Detection Limits (MDL) and Reporting Limits (RL)	5-18
Sacramento -5-13	Method 1668 Method Detection Limits (MDL) and Reporting Limits (RL)	5-19
Sacramento -5-14	Method 1653 Method Detection Limits (MDL) and Reporting Limits (RL)	5-20
Sacramento -5-15	Method 1613B Dioxins and Furans Method Detection Limits (MDL) and Reporting Limits (RL)	5-21
Sacramento -5-16	Method 551 Dioxins and Furans Method Detection Limits (MDL) and Reporting Limits (RL)	5-22
Sacramento -5-17	Chemical Degradates Method Detection Limits (MDL) and Reporting Limits (RL)	5-23

List of Tables (Continued)

Table No.	Title	Section - Page
Sacramento -5-18	Method 8280 Dioxins and Furans Method Detection Limits (MDL) and Reporting Limits (RL)	5-24
Sacramento -5-19	Method 8280A Dioxins and Furans Method Detection Limits (MDL) and Reporting Limits (RL)	5-25
Sacramento -5-20	8290 Dioxins and Furans Method Detection Limits (MDL) and Reporting Limits (RL)	5-26
Sacramento -5-21	Method 8321 Method Detection Limits (MDL) and Reporting Limits (RL)	5-28
Sacramento -5-22	Method 8321A Method Detection Limits (MDL) and Reporting Limits (RL)	5-29
Sacramento -5-23	Tetrazene by Method 8331 Method Detection Limits (MDL) and Reporting Limits (RL)	5-30
Sacramento -5-24	Method 200.8 ICP MS Detection Limits (MDL) and Reporting Limits (RL)	5-31
Sacramento -5-25	Mercury Method Detection Limits (MDL) and Reporting Limits (RL)	5-32
Sacramento -5-26	Method 6010B ICP Trace Method Detection Limits (MDL) and Reporting Limits (RL)	5-33
Sacramento -5-27	Method 6010B ICP Method Detection Limits (MDL) and Reporting Limits (RL)	5-34
Sacramento -5-28	Method 6020 ICP MS Method Detection Limits (MDL) and Reporting Limits (RL)	5-35
Sacramento -5-29	Graphite Furnace Metals Method Detection Limits (MDL) and Reporting Limits (RL)	5-37
Sacramento -5-30	Method 1664 Oil and Grease Method Detection Limits (MDL) and Reporting Limits (RL)	5-38
Sacramento -5-31	General Chemistry Method Detection Limits (MDL) and Reporting Limits (RL)	5-39
Sacramento -6-1	Performance Evaluation Sample Program	6-3

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Facility Appendix
Sacramento Laboratory

Section Sacramento-1

Organizational
Chart

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Quanterra - West/Sacramento

Regional General Manager
Roger Freize
78

Regional Financial Manager/ Capacity Manager
Tracy Gingerich

Laboratory Manager/ Technical Director
Eric Redman
76

Joe Schairer, AA/HR Coordinator

Technical Director Air Toxics
Patrick Rainey
4

Brian Thompson
Steve Valmores
Marilyn Toomey

Manager of Advanced Technology Operations
Karla Buechler
23

Dioxin Prep - 7
Open - AL

Cyndi Westbrook
Marc Onishi
Boyd Harling
Kevin Sanchez
Ritesh Jivan
Betty Damron
Hoa Nguyen

Dioxin Instruments - 11
Clark Pickell - AL

Andre Algazi
Saleh Arghestani
Maricel Baquerfo
Mike Flournoy
Mark Grandfield
Adrian Messecar
Teri Stone
Keith Sturgeon
Steve Rogers
Rosie Laster

Energetics/Specialty Chemicals - 4

Mike Filigenzi
Dennis Gall
Hanh Nguyen
Robert Hrabak
Open

Emily Uebelhoer - Asst Ops Mgr
Semi Voas 9

Lisa Stafford – Organic systems lead
Organic Prep
Karen Mason
Jonathan Reed
George Burkett
Semi VOAs
Donald Taylor
Jon Edmondson
Marcia Reed
Pam Niya

Volatiles
Wayne Halozan - AL 6

Yun Pan
Kyle Stephens
Chris Jenkins
Mike Lucchesi
Karen Yee

Metals
John Barnett AL 4

David Allbucker
Allan Wong
Mei Lai
Open

Gen Chem
Josie Jones AL 4

Tuan Nguyen
Mark Frey
Lesa Waldron

Manager of Customer Service
Dave Herbert
23

Project Management

Advanced Tech - 3
Terry Wilson - AL

Nanny Estrada
Jill Kellmann
Open
Open (P/T)

Air Toxics – 3
Kathy Gill - AL

Kari Filigenzi
Robert Weidenfeld

Environmental Services – 5
Beth Kelly - AL

Hilo Lipi
Diana Brooks
Jon Gildersleeve
Bonnie McNeill

Data Management - 7
Open - AL

Rob Alai
Ayla Douglas
Kelly Gonyea
Vicki Herd (P/T)
Joe Lee
Kim Weber
Doug Winter

Internal Support - 4
Ray Lucero - AL

Marcela Garcia
Cheryl Hytock
Richard Kester
Open

Quality Assurance Manager
Pam Schemmer
2

Gail Celaschi
Open (P/T)

Proprietary Information
For Internal Use Only

TABLE 1-1
SACRAMENTO LABORATORY
Key Personnel List

ASSOCIATE	JOB TITLE	DEGREE	DATE OF HIRE
Roger Freize, II	Western Regional Manager	M.B.A. B.S., Petroleum Engineering	5-06-85
Eric Redman	Laboratory Manager, Technical Director	M.S., Chemical Physics B.S., Chemistry and Physics	8-10-88
Dave Herbert	Systems Manager	B.S. Biochemistry	5-22-89
Pamela Schemmer	Quality Assurance Manager	B.S., Chemistry	2-08-99
Wayne Halozan	Area Leader, VOA Instruments	B.A. Chemistry	5-17-99
Elizabeth R. Stafford	Organics Systems Specialist	B.S., Chemistry	9-08-87
John Barnett	Area Leader, Metals	B.A., Environmental Biology	6-01-80
Patrick G. Rainey	Technical Director, Air Toxics NELAC Deputy Technical Director	B.S., Environmental Toxicology	10-20-88
Karla S. Buechler	Operations Manager/Deputy Technical Director	B.S., Biochemistry	9-29-86
Clark Pickell	Area Leader, Advanced Technology Instruments	B.S., Physics	10-07-91
Terry A. Wilson	Area Leader, Project Management	B.A., Biology	7-11-89
Kathy Gill	Area Leader, Project Management	B.A., Biology	10-88
Elizabeth (Beth) Kelly	Area Leader, Project Management	B.A., Chemistry	8-88
Emily R. Uebelhoer	Assistant Operations Manager/Deputy Technical Director	Lexington Christian Academy	1-05-99
Ray Lucero	Area Leader, Support Services		10-13-86
Josie Jones	Area Leader, General Chemistry	B.S., Chemistry	11-25-85

Facility Appendix
Sacramento Laboratory

Section Sacramento-2

Instrument
List

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TABLE 2-1
SACRAMENTO LABORATORY
Instrument List

Instrument Type	Manufacturer	Model-Serial #	Purchase Date	Autosampler
GC/MS	Finnigan	INCOS 50-IN276	1989	Yes
	Finnigan	INCOS 50-IN354	1988	Yes
	Finnigan	INCOS 50	1988	Yes
	Finnigan	INCOS 50	1988	Yes
	Hewlett-Packard	HP 5970-2905A11727	1989	Yes
	Hewlett-Packard	HP 5970-2905A11646	1989	Yes
	Hewlett-Packard	HP 5970-2905A11792	1989	Yes
	Hewlett-Packard	HP 5970-3004A12561	1991	Yes
	Hewlett-Packard	HP 5970-2905A12019	Unknown	Yes
	Hewlett-Packard	HP 6890-DE00020255	1998	Yes
	Hewlett-Packard	HP 6890-US00021334	1998	Yes
	Hewlett-Packard	HP 6890-US00021417	1998	Yes
	Hewlett-Packard	HP 5973-US80221311	Lease	Yes
HRGC/LRMS	Hewlett-Packard	HP 6890-US80221311	1999	Yes
	Hewlett-Packard	HP 6890 US82321717	6/18/99	Yes
	Hewlett-Packard	HP 6890 US82321724	6/24/99	Yes
	Finnigan	4500	1984	Yes
	Finnigan	4500	1986	Yes
	Finnigan	4500	1986	Yes
HRGC/HRMS	Fisons	VG70/250S-7054	1988	Yes
	Fisons	VG70/VSE-6032	1989	Yes
	Fisons	VG70/VSE-7088	1990	Yes
	Fisons	Ultima Autospec-M421	1992	Yes
	Fisons	Ultima Autospec U-S176U	1998	Yes
GPC	ABC GPC Systems	1002B	-----	Yes
Data System	Finnigan	Nova 4	1981	No

Instrument Type	Manufacturer	Model-Serial #	Purchase Date	Autosampler
	Finnigan	Nova 4	1981	No
	Fisons	VAX 3600	1992	No
LC/MS	Finnigan	TSQ 700-13967	1987	Yes
	Finnigan	TSQ 700-TS363	1989	Yes
GC-ECD/TSD	Varian	V 3400-5458	1988	Yes
GC-FID/FID	Hewlett-Packard	6890-US00000311	1997	Yes
	Hewlett-Packard	6890-US00006455	1997	Yes
GC-ECD/ECD	Hewlett-Packard	6890-US00001087	1997	Yes
	Hewlett-Packard	6890-US00006442	1997	Yes
	Hewlett-Packard	6890-US00006441	1997	Yes
	Hewlett-Packard	6890-US00006438	1997	Yes
	Varian	V 3400-4152	1987	No
	Varian	V 3400-4139	1987	Yes
	Varian	V 3400-10220	1990	Yes
	Varian	V 3400-10221	1990	Yes
GC/FID	Varian	V 3400-1170	1985	No
GC-ELCD/ELCD	Hewlett-Packard	6890-US00006458	1997	Yes
	Hewlett-Packard	6890-US00006465	1997	Yes
GC-PID/PID/FID	Hewlett-Packard	6890-US00006463	1997	Yes
	Hewlett-Packard	6890-US00006440	1997	Yes
	Hewlett-Packard	6890-US00006439	1997	Yes
GC-FPD/TSD	Varian	3300-2095	1985	Yes
	Varian	3740-81041622	1984	Yes
HPLC	Varian	V 5020-7243	----	No
	Varian	V 5500-0640	1984	Yes
	Varian	V 5560-1315	1986	Yes
	Varian	V 5500-1618	1988	Yes
	Hewlett-Packard	1050/1046A	1999	Yes

TABLE 2-1
SACRAMENTO LABORATORY
Instrument List
(Continued)

Instrument Type	Manufacturer	Model-Serial #	Purchase Date	Autosampler
ICP	Thermo Jarrell Ash	9000-57483	1986	Yes
	Thermo Jarrell Ash	61e-44190	1990	Yes
ICP/Trace	Thermo Jarrell Ash	61E/275490	1994	Yes
ICP/MS	Perkin-Elmer	ELAN 6000-051950460	1994	Yes
	Perkin-Elmer	ELAN 6000-4719801	1998	Yes
GFAA	Perkin-Elmer	ZEEMAN 5100Z-6613	1989	Yes
	Perkin-Elmer	ZEEMAN 5100Z-6751	1988	Yes
	Perkin-Elmer	ZEEMAN 5100Z-6419	1990	Yes
Cold-Vapor Analyzers	Leeman	PS200-0270	1989?	Yes
	Leeman	PS200 II-HG-8008	1998	Yes
Wet Chem	Lachat	Quick Chem	1986	Yes
	Lachat	Model AE	1990	Yes
	Mitsubishi	TOX-10Sigma #1	1991	No
	Mitsubishi	TOX-10Sigma #2	1993	No
	Dohrmann	DC 190 - TOC #2	1992	Yes
	OI Corp	700-TOC #1	1987	Yes
	Radiometer	T-T85-Autotitrator	1991	Yes
	Dionex	20101-IC System 1	1986	Yes
	Dionex	DX100-ICDX	1993	Yes
	Dionex	IC Cr+6	1993	Yes
	Milton Roy	21D - Spec.	1991	No
	Milton Roy	21D - Spec.	1992	No

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Facility Appendix
Sacramento Laboratory

Section Sacramento-3

Standard
Operating
Procedures

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TABLE 3-1
SACRAMENTO LABORATORY
Standard Operating Procedures List

SOP No.	Rev. #	Title	Date
CORP-GC-0001	5	Gas Chromatographic Analysis Based on Method 8000B,8021A,8082 & 8151A, SW-846	4/8/99
CORP-GC-0001SAC	4.1	Gas Chromatographic Analysis Based on Method 8000B, 8021B,8081A, 8082 & 8151A,SW-846	10/25/98
CORP-IP-0001	0	Acid Digestion of Waters & Soils, CLP SOW ILMO3.0	5/28/98
CORP-IP-0002	1	Acid Digestion of Soils, SW846 Method 3050B	2/2/98
CORP-IP-0002SAC	1.1	Acid Digestion of Soils, SW846 Method 3050B	10/15/98
CORP-IP-0003	0	Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods	12/9/95
CORP-IP-0003SAC	0.1	Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods	10/15/98
CORP-IP-0004	0	Toxicity Characteristic Leaching Procedure & synthetic Precipitation Leaching Procedure	9/14/96
CORP-IP-0004CF	0	Toxicity Characteristic Leaching Procedure &Synthetic Precipitation Leaching Procedure	4/8/99
CORP-IT-0001	0	Software and Hardware Change Management	7/21/97
CORP-IT-0002	0	Information Technology Service Request	10/3/97
CORP-IT-0005	0	LIMS	7/22/97
CORP-IT-0007	0	Software Testing, Validation, and Verification	6/13/97
CORP-MS-0001	2	GC/MS Analysis Based on Methods 8270C & 625	2/2/98
CORP-MS-0001SAC	2.1	GC/MS Analysis Based on Methods 8270C & 625	9/9/98
CORP-MS-0002	2	Determination of Volatile Organics by GC/MS Based on Method 8260B, 624, and 524.2	2/2/98
CORP-MS-0002CF	2	Determination of Volatile Organics by GC/MS based on Method 8260B, 624 and 524.2	5/1/98
CORP-MS-0002SAC	2.1	Determination of Volatile Organics by GC/MS Based on Method 8260B, 624, 524.2	3/3/97
CORP-MT-0001	2	ICP-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW846 Method 6010B & EPA method 200.7	2/2/98
CORP-MT-0001SAC	2.1	ICP-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Method 6010B & EPA method 200.7	9/8/98
CORP-MT-0002	1	ICP-Atomic Emission Spectroscopy, Method 200.7 CLP-M, SOW ILMO3.0	10/6/95
CORP-MT-0002SAC	1.1	ICP-Atomic Emission Spectroscopy, Method 200.7 CLP-M, SOW ILMO3.0	1/12/96
CORP-MT-0003	1	GFAA Spectroscopy, SW-846 Method 7000A & MCAWW 200 Series Methods	10/6/95
CORP-MT-0003SAC	1.2	Graphite Furnace Atomic Absorption Spectroscopy, SW-846 Method 7000A & MCAWW 200 Series Methods	10/15/98

TABLE 3-1
SACRAMENTO LABORATORY
Standard Operating Procedures List

SOP No.	Rev. #	Title	Date
CORP-MT-0004	1	GFAA Spectroscopy Method, 2XX.X CLP-M, SOW ILMO3.0	10/6/95
CORP-MT-0004SAC	1.1	GFAA Spectroscopy Method, 2XX.X CLP-M, SOW ILMO3.0	1/8/99
CORP-MT-0005	1	Preparation & Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A & MCAWW 245.1	3/6/96
CORP-MT-0005CF #1	1	Preparation & Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A & MCAWW 245.1	8/1/96
CORP-MT-0005CF 2	1	Preparation & Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A & MCAWW 245.1	8/1/96
CORP-MT-0005CF 3	1	Preparation & Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A & MCAWW 245.1	8/1/96
CORP-MT-0005CF 4	1	Preparation & Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A & MCAWW 245.1	8/1/96
CORP-MT-0005CF1	1	Preparation & Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A & MCAWW 245.1	8/1/96
CORP-MT-0006	0	Preparation & Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, Method 245.1 CLP-M, SOW ILMO3.0	3/5/96
CORP-MT-0007	1	Preparation & Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SW846 7471A & MCAWW 245.5	3/5/96
CORP-MT-0007CF	1	Preparation & Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SW846 7471A & MCAWW 245.5	8/1/96
CORP-MT-0008	0	Preparation & Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, Method 245.5 CLP-M, SOW ILMO3.0	3/5/96
CORP-MT-0008CF1	0	Preparation & Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, Method 245.5 CLP-M, SOW ILMO3.0	8/1/96
CORP-MT-0008CF2	0	Preparation & Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, Method 245.5 CLP-M, SOW ILMO3.0	8/1/96
CORP-MT-0008CF3	0	Preparation & Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, Method 245.5 CLP-M, SOW ILMO3.0	8/1/96

TABLE 3-1
SACRAMENTO LABORATORY
Standard Operating Procedures List

SOP No.	Rev. #	Title	Date
CORP-OP-0001	3	Extraction & Cleanup of Organic Compounds from Waters & Soils, Based on SW-846 3500 Series, 3600 Series, 8151A & 600 Series Methods	2/3/98
CORP-OP-0001CF	3	Extraction & Cleanup of Organic compounds form Waters & Soils, Based on SW-846 3500 Series, 3600 Series, 8151A & 600 Series	4/9/99
CORP-OP-0001SAC	3.1	Extraction & Cleanup of Organic Compounds from Waters & Soils, Based on SW-846 3500 series, 3600 Series, 8151A, and 600 Series methods	9/25/98
CORP-QA-0001	3	Quality Testing of Solvents, Acids, & Reagents (Quality-Related Items (QRI) Program)	2/8/99
CORP-QA-0002	0	QuantIMS Reference Data Creation and Maintenance	1/27/95
CORP-QA-0004	2	INDEPENDENT QA DATA REVIEW	10/12/98
CORP-QA-0010	1	NONCONFORMANCE AND CORRECTIVE ACTION	8/13/97
CORP-QA-0012	1	Selection and Evaluation of Subcontractor Laboratories	2/5/98
CORP-QA-0013	1	EMPLOYEE ORIENTATION AND TRAINING	2/8/99
CORP-QA-0014	0	LABORATORY INTERNAL SYSTEMS EVALUATION	6/30/97
CORP-WC-0001	0	Total Organic Halides in Waters by SW-846 Method 9020B	4/28/96
CORP-WC-0002	1	Determination of Solids in Waters and Wastes	6/28/96
CORP-WC-0002SAC	1.1	determination of all Types of Residue in Waters, Wastes, and Solid Samples	10/9/98
CORP-WC-0003	0	HEM/SGT-HEM by Method 1664 (Formerly Oil & Grease/TPH)	3/5/96
ITAS-IT-MT-0005	0	Metals Analysis By GFAA Spectrophotometer by SW846 7000 Series & EPA 200 Series Methods	9/18/93
LM-CAL-3066	1	Method 1613 Rev.A- Tetra-through Octa- Chlorinated Dioxins & Furans by Isotope Dilution HRGC/HRMS	5/10/94
SAC--ID-0005CF1	0	Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS	8/24/99
SAC-GC-0006	0	Determination of White Phosphorous in Soil by Gas Chromatogaphy with flame-Photometric Detector (GC/FPD)	2/24/97
SAC-GC-0007	1	Gas Chromatographic Analysis Based on Method 8000B,8051B,SW-846 UpdateIII & CALIF. LUFT Manual, 10/89	10/15/98
SAC-GC-0009	0	Gas Chromatographic Analysis Based on Method 8000B,8151A, Modified, 8141, & 8310, SW-846	9/2/98
SAC-GC-0010	0	Gas Chromatographic Analysis of Prepared Extracts for Total Chromatographable Organics (TCO), Semivolatile & Volatile Fractions	3/17/98
SAC-ID-0005	4	Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS	9/25/98

TABLE 3-1
SACRAMENTO LABORATORY
Standard Operating Procedures List

SOP No.	Rev. #	Title	Date
SAC-ID-0005CF1	0	Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS	6/15/99
SAC-ID-0005CF2	0	Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS	8/24/99
SAC-ID-0008	3	Analysis of Polychlorinated Dioxins/Furans by Low Resolution GC/MS	2/14/97
SAC-ID-0011	1	Extraction & Analysis of Polychlorinated Dibenzo-P-Dioxins & Dibenzofurans by DFLM01.0, Including Revision DFLM01.1 (9/91) & Method 8280A	10/15/98
SAC-ID-0012	1	Responsibilities of a Spike Witness- High Resolution Dioxins	10/15/98
SAC-ID-0013	0	PCB Analysis by HRGC/HRMS	6/30/99
SAC-IP-0003	1	Waste Extraction Test for CCR STLC (Citrate Buffer/Deionized Water)	7/9/99
SAC-IP-0006	0	Method for the Determination of Particulate Matter in the Atmosphere as TSP or PM10 (high volume method)	9/13/96
SAC-IP-0006CS	1	Determination of Particulate Matter in the Atmosphere as Total Suspended Particulates or PM10 (High-volume Method)	4/17/9[illegible]
SAC-IP-0007	0	Metals Preparation of emissions Test Method 29	7/29/96
SAC-IP-0008	1	Metals Matrix Matching for Analysis of Dissolved Metals	9/25/98
SAC-IT-0001	1	VAX and SEED LIMS BACKUP	9/3/98
SAC-LC-0001	6	The Extraction & Analysis of Nitroaromatics, nitramines, & Specialty Explosives in H20 & Soil by HPLC with an UV Detector & LC with a Thermospray Interfaced to a MS	10/15/98
SAC-MS-0004	1	Preventative Maintenance for VG-70 GC/MS Systems	10/15/98
SAC-MT-0001	1	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry	9/25/98
SAC-MT-0002	1	Analysis of Bromide by ICP/MS (GLP Protocol).	1/8/99
SAC-OP-0006	1	Preparation & Extraction of Semi-Volatiles on PUF/XAD-2 Resin Samples for GC/MS analysis	3/1/1196
SAC-OP-0006CS	0	Preparation & Extraction of Semi-volatile Organic Constituents on PolyUrethane Foam/XAD-2 Resin Samples for GC/MS	4/4/96
SAC-OP-0007	0	Method TO-4(modified): Extraction of Organochlorine Pesticides for GC/ECD Analysis	2/19/97
SAC-OP-0007 CF	0	Method TO-4(modified): Extraction of Organochloride Pesticides for GC/ECD ananlysis	6/1/98

TABLE 3-1
SACRAMENTO LABORATORY
Standard Operating Procedures List

SOP No.	Rev. #	Title	Date
SAC-OP-0010	0	Extraction of Pentachlorophenol & 2,3,4,6-Tetrachlorophenol in Water & Soil by method SW-8151A	6/2/98
SAC-OP-0011	0	Cleaning of Glassware (Organics)	3/5/98
SAC-OP-0012	1	Daily setup Procedure &Maintenance of the ABC 1002B GPC Sampler Processing Unit	9/25/98
SAC-OP-0013	1	Determination of Percent Moisture	9/25/98
SAC-OP-4177	1	Despatch VRE2-35-1E Oven Operation	9/4/98
SAC-QA-0001	5	BUILDING SECURITY	9/4/98
SAC-QA-0002	1	Procedure for the set-up. maintenace and analysis of holding blanks for volatile refrigerators	9/8/98
SAC-QA-0003	6	Sample Receipt and Procedures	9/25/98
SAC-QA-0004	2	Maintenance & Calibration check of Fixed & Adjustable Volume Autopipettors	9/4/98
SAC-QA-0005	2	Temperature Monitoring & Corrective Actions For Refrigerators & Freezers	4/10/98
SAC-QA-0006	1	Method Detection Limits (MDL) & Instrument Detection Limits (IDL)	9/4/98
SAC-QA-0007	5.1	Bottles and Cooler Preparation	10/15/98
SAC-QA-0009	1.1	Document Archiving	9/25/98
SAC-QA-0014	0	Monitoring of Reagent-Grade Laboratory Water	12/18/97
SAC-QA-0015	0	Standards and QC Solutions Preparation - Metals	10/15/98
SAC-QA-0016	1	Equipment Monitoring & Thermometer Calibration	7/22/98
SAC-QA-0017	0	Standards & Reagent Preparation & Quality Control Check Procedures	10/9/98
SAC-QA-0041	2	Calibration & Calibration Check of Balances	9/4/98
SAC-WC-0009	0	Determination of Anions by Ion Chromatography (EPA Method 300.0-Part A, SW-846/Method 9056)	10/9/98
SAC-WC-0011	0	Determination of Hexavalent Chromium emissions from Stationary Sources by Ion Chromatography (method Title 40 CFR, Part 266, Appendix IX, Section 3.2 Modified), and Method CARB 425	9/27/96
SAC-WC-0012	0	Determination of Hydrogen Halides & Halogen Emissions from Stationary Sources by Ion Chromatography (Method 26, 26A and method 9057)	9/27/96
SAC-WC-0014	0	Sulfide by EPA 376.2- Modified	7/9/99
SAC-WC-0015	0	Determination of Cyanides (All Types) Using Manual Distillation, Followed by Automated Colorimetry	10/9/98
SAC-WC-0016	0	Total Organic Carbon in Water by Oxidation Method-Method 9060 Modified, EPA 415.1, SM 5310C	4/8/99

TABLE 3-1
SACRAMENTO LABORATORY
Standard Operating Procedures List

SOP No.	Rev. #	Title	Date
SAC-WC-0020	0	Determination of Hexavalent Chromium by Manual Colorimetric Method	10/9/98
SAC-WC-0027	1	Turbidity (Nephelometric Method)	7/9/99
SAC-WC-0028	1	Determination of Alkalinity, Conductivity and pH	7/9/99
SAC-WC-0032	1	Hardness, Total (MG/L as CaCO3- Method 130.2 (Titrimetric EDTA)	7/9/99
SAC-WC-0033	1	Fluoride by Electrode	7/9/99
SAC-WC-0040	1	Determination of Chemical Oxygen Demand	7/9/99
SAC-WC-0041	1	Ignitability	7/9/99
SAC-WC-0044	1	EPA Method 9045B pH soils	12/16/97
SAC-WC-0049	0	Deionized Water Leaching Procedure for General Chemistry Analyses	3/11/98
SAC-WC-0050	0	Preparation and Analysis of Nitrocellulose in Aqueous and Soil/Sediment Samples by Colorimetric Autoanalyzer	7/14/99
SAC-WC-0051	0	Basic Operation of Lachat Quikchem Automated Ion Analyzer (Automated colorimetry)	10/15/98
SAC-WC-0052	0	Pipette & Burette Washing Procedure for Inorganics Analyses (Metals & Gen. Chem.)	10/15/98
SAC-WC-0105	2	Ammonia as N (Lachat Autoanalyzer)	7/9/99
SANA-OP-0003	0	Extraction of Fuel Class Hydrocarbons (BLS-191)	4/14/95

Facility Appendix

Sacramento Laboratory

Section Sacramento-4

Analytical Methods

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TABLE 4-1
SACRAMENTO LABORATORY
Organic Preparation and Cleanup Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Volatile organics (VOAs)	Aqueous	524.2		SW846 5030B SW846 1311	-
	Solid	-	-	SW846 5035 SW846 1311	-
	Waste	-	-	SW846 5035 SW846 3585 SW846 1311	-
	Biological	-	-	-	-
	Air	-	-	-	-
Base neutrals and acids (BNAs)	Aqueous	-	-	SW846 3510C SW846 3520C SW846 1311 Calif. CAM-WET	EPA 23 SW3542
	Solid	-	-	SW846 3540C SW846 3550B SW846 1311 Calif. CAM-WET	-
	Waste	-	-	SW846 3580A SW846 1311 Calif. CAM-WET	-
	Biological	-	-	SW846 3550B	-
	Air	-	-	-	EPA 23 T0-13 SW3542

TABLE 4-1
SACRAMENTO LABORATORY
Organic Preparation and Cleanup Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Organochlorine Pesticides	Aqueous	-	-	SW846 3510C SW846 3520C SW846 3600C SW846 1311 Calif. CAM-WET	
	Solid	-	-	SW846 3540C SW846 3550B SW846 3600C SW846 1311 Calif. CAM WET	-
	Waste	-	-	SW846 3580A SW846 3600 SW846 1311 Calif. CAM WET	-
	Biological	-	-	SW846 3550B	-
	Air	-	-	-	TO-4 TO-10
PCBs	Aqueous	-	-	SW846 3510C SW846 3520C SW846 3600C SW846 1311 Calif. CAM WET	EPA 23
	Solid	-	-	SW846 3540C SW846 3550B SW846 3600C SW846 1311 Calif. CAM WET	-
	Waste	-	-	SW846 3580A SW846 3600C SW846 1311 Calif. CAM WET	-
	Biological	-	-	-	-
	Air	-	-	-	EPA 23 TO-4 TO-10

TABLE 4-1
SACRAMENTO LABORATORY
Organic Preparation and Cleanup Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Petroleum Hydrocarbons	Aqueous	-	-	SW846 5030B SW846 3510C SW846 3520C GRO/DRO[1]	-
	Solid	-	-	SW846 5035 SW846 3540C SW846 3550B GRO/DRO[2]	-
	Waste	-	-	SW846 3580 GRO/DRO[3]	-
	Biological	-	-	-	-
	Air	-	-	-	-
EDB/DBCP	Aqueous	EPA 504.1	EPA 504.1	SW846 8011	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Nitroaromatics and Nitramines	Aqueous	-	-	SW846 8330 SW846 3535	-
	Solid	-	-	SW846 8330	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	SW846 8330 SW846 8321

[1] GRO/DRO represent the broad category of methods used to analyze samples for Gasoline Range Organics and Diesel Range Organics. For Quanterra (WSAC), these methods include California LUFT methods, Washington TPH methods, Arizona TPH, and Alaska TPH methods.
[2] GRO/DRO represent the broad category of methods used to analyze samples for Gasoline Range Organics and Diesel Range Organics. For Quanterra (WSAC), these methods include California LUFT methods, Washington TPH methods, Arizona TPH, and Alaska TPH methods.
[3] GRO/DRO represent the broad category of methods used to analyze samples for Gasoline Range Organics and Diesel Range Organics. For Quanterra (WSAC), these methods include California LUFT methods, Washington TPH methods, Arizona TPH, and Alaska TPH methods.

TABLE 4-1
SACRAMENTO LABORATORY
Organic Preparation and Cleanup Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Herbicides	Aqueous	-	-	SW846 8151A SW846 1311 Calif. CAM WET	-
	Solid	-	-	SW846 8151A SW846 1311 Calif. CAM WET	-
	Waste	-	-	SW846 8151A SW846 1311	-
	Biological	-	-	-	-
	Air	-	-	-	-
Polynuclear aromatic hydrocarbons	Aqueous	-		SW846 3510C SW846 3520C SW846 1311	CARB 429
	Solid	-	-	SW846 3540C SW846 3550B SW846 1311	-
	Waste	-	-	SW846 1311	-
	Biological	-	-	SW846 3550B	-
	Air	-	-	-	CARB 429
White Phosphorus	Aqueous	-	-	SW846 3510C	-
	Solid	-	-	SW846 3550B	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Tetrazene	Aqueous	-	-	SW846 8331	-
	Solid	-	-	SW846 8331 SW846 1311 Calif. CAM WET	-
	Waste*	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-1
SACRAMENTO LABORATORY
Organic Preparation and Cleanup Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Chemical Warfare Degradates	Aqueous	-	-	SW846 3510C	-
	Solid	-	-	SW846 8330	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Thiodiglycol	Aqueous	-	-	SW846 8330	-
	Solid	-	-	SW846 8330	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Picric Acid	Aqueous	-	-	SW846 8330	-
	Solid	-	-	SW846 3550B	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Organosulfur Degradates	Aqueous	-	-	SW846 3510C	-
	Solid	-	-	SW846 8330	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Chlorinated Phenolics	Aqueous	-	-	EPA 1653	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-1
SACRAMENTO LABORATORY
Organic Preparation and Cleanup Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Polychlorinated Biphenyl Congeners	Aqueous	-	-	SW846 3510C	-
	Solid	-	-	SW846 3540C	-
	Waste	-	-	SW846 3580	-
	Biological	-	-	SW846 3540C	-
	Air	-	-	-	SW846 0023A

TABLE 4-1
SACRAMENTO LABORATORY
Organic Preparation and Cleanup Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans	Aqueous	EPA 1613B NCASI 551	EPA 1613B NCASI 551	SW846 8290 SW846 8280A DFLM01.1 SW846 1311 Calif. CAM WET	SW846 0023A EPA 23
	Solid	-	EPA 1613B NCASI 551	SW846 8290 SW846 8280A DFLM01.1 SW846 1311 Calif. CAM WET	-
	Waste	-	-	SW846 8290 SW846 8280A DFLM01.1 SW846 1311 Calif. CAM WET	-
	Biological	-	-	SW846 8290 SW846 8280A	-
	Air	-	-	-	TO-9 SW846 0023A

TABLE 4-2
SACRAMENTO LABORATORY
Organic Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Volatile organics (VOAs)	Aqueous	EPA 524.2	-	SW846 8260B SW846 8021B	-
	Solid	-	-	SW846 8260B SW846 8021B	-
	Waste	-	-	SW846 8260B SW846 8021B	-
	Biological	-	-	-	-
	Air	-	-	-	E1600-8
Base neutrals and acids (BNAs)	Aqueous	-	-	SW846 8270C	TO-13 EPA 23
	Solid	-	-	SW846 8270C	-
	Waste	-	-	SW846 8270C	-
	Biological	-	-	SW846 8270C	-
	Air	-	-	-	TO-13 EPA 23
Organochlorine Pesticides	Aqueous	-	-	SW846 8081A	TO-4 TO-10
	Solid	-	-	SW846 8081A	-
	Waste	-	-	SW846 8081A	-
	Biological	-	-	SW846 8081A	-
	Air	-	-	-	TO-4 TO-10
PCB Aroclors	Aqueous	-	EPA 608	SW846 8082	EPA 23 TO-4 TO-10
	Solid	-	-	SW846 8082	-
	Waste	-	-	SW846 8082	-
	Biological	-	-	-	-
	Air	-	-	-	EPA 23 TO-4 TO-10

TABLE 4-2
SACRAMENTO LABORATORY
Organic Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Petroleum Hydrocarbons	Aqueous	-	-	Calif LUFT GRO/DRO[4]	-
	Solid	-	-	Calif. LUFT GRO/DRO[4]	-
	Waste	-	-	Calif. LUFT GRO/DRO[4]	-
	Biological	-	-	-	-
	Air	-	-	-	-
EDB/DBCP	Aqueous	EPA 504.1	EPA 504.1	SW846 8011	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Nitroaromatics and Nitramines	Aqueous	-	-	SW846 8330 SW846 8321	-
	Solid	-	-	SW846 8330 SW846 8321	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	SW846 8330 SW846 8321
Herbicides	Aqueous	-	-	SW846 8151A	-
	Solid	-	-	SW846 8151A	-
	Waste	-	-	SW846 8151A	-
	Biological	-	-	-	-
	Air	-	-	-	-

[4] GRO/DRO represent the broad category of methods used to analyze samples for Gasoline Range Organics and Diesel Range Organics. For Quanterra WSAC, these methods include California LUFT methods, Washington, Arizona, and Alaska TPH methods, which are specifically included by reference.

TABLE 4-2
SACRAMENTO LABORATORY
Organic Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Polynuclear aromatic hydrocarbons	Aqueous	-	-	SW846 8310 CARB 429	CARB 429
	Solid	-	-	SW846 8310 CARB 429	-
	Waste	-	-	SW846 8310 CARB 429	-
	Biological	-	-	CARB 429	-
	Air	-	-	-	CARB 429
White Phosphorus	Aqueous	-	-	SW846 8141	-
	Solid	-	-	SW846 8141	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Tetrazene	Aqueous	-	-	SW846 8331	-
	Solid	-	-	SW846 8331	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Chemical Warfare Degradates	Aqueous	-	-	SW846 8321	-
	Solid	-	-	SW846 8321	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Thiodiglycol	Aqueous	-	-	SW846 8330	-
	Solid	-	-	SW846 8330	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-2
SACRAMENTO LABORATORY
Organic Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Picric Acid	Aqueous	-	-	SW846 8321	-
	Solid	-	-	SW846 8321	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Organosulfur Degradates	Aqueous	-	-	SW846 8270C	-
	Solid	-	-	SW846 8270C	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Chlorinated Phenolics	Aqueous	-	-	EPA 1653	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Polychlorinated Biphenyl Congeners	Aqueous	-	-	EPA 1668	-
	Solid	-	-	EPA 1668	-
	Waste	-	-	EPA 1668	-
	Biological	-	-	EPA 1668	-
	Air	-	-	-	EPA 1668
Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans	Aqueous	EPA 1613B NCASI 551	EPA 1613B NCASI 551	SW846 8290 SW846 8280A DFLM01.1	SW846 0023A EPA 23
	Solid	-	EPA 1613B NCASI 551	SW846 8290 SW846 8280A DFLM01.1	-
	Waste	-	-	SW846 8290 SW846 8280A DFLM01.1	-
	Biological	-	-	SW846 8290 SW846 8280A	-
	Air	-	-	-	TO-9 SW846 0023A

TABLE 4-3
SACRAMENTO LABORATORY
Metals Preparation Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CLP	CAA
ICP Metals	Aqueous	-	-	SW846 3005A SW846 3010A SW846 1311 SW846 1312 (W) Calif. CAM WET	SW846 0060 EPA 29
	Solid	-	-	SW846 3050B SW846 1311 SW846 1312 (W) Calif. CAM WET	-
	Waste	-	-	SW846 3050B SW846 1311 SW846 1312 (W) Calif. CAM WET	-
	Biological	-	-	SW846 3050B	-
	Air	-	-	-	SW846 0060 EPA 29
Mercury	Aqueous	EPA 245.1	EPA 245.1	SW846 7470A SW846 1311 SW846 1312 (W) Calif. CAM WET	SW846 0060
	Solid	-	-	SW846 7471A SW846 1311 SW846 1312 (W) Calif. CAM WET	-
	Waste	-	-	SW846 7471A SW846 1311 SW846 1312 (W) Calif. CAM WET	-
	Biological	-	-	SW846 7471A	-
	Air	-	-	-	SW846 0060 EPA 29

TABLE 4-3
SACRAMENTO LABORATORY
Metals Preparation Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CLP	CAA
Chromium (GFAA)	Aqueous	-	-	-	SW846 0060
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	SW846 0060
Lead (GFAA)	Aqueous	-	-	SW846 7421	SW846 0060
	Solid	-	-	SW846 7421	-
	Waste	-	-	SW846 7421	-
	Biological	-	-	-	-
	Air	-	-	-	SW846 0060 EPA PM-10
Selenium (GFAA)	Aqueous	-	-	SW846 7740 SW846 1311 SW846 1312 (W) Calif. CAM WET	SW846 0060
	Solid	-	-	SW846 7740 SW846 1311 SW846 1312 (W) Calif. CAM WET	-
	Waste	-	-	SW846 7740 SW846 1311 SW846 1312 (W) Calif. CAM WET	-
	Biological	-	-	-	-
	Air	-	-	-	SW846 0060 EPA PM-10
Thallium (GFAA)	Aqueous	-	-	SW846 7841	SW846 0060
	Solid	-	-	SW846 7841	-
	Waste	-	-	SW846 7841	-
	Biological	-	-	-	-
	Air	-	-	-	SW846 0060 EPA PM-10

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Aluminum	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-		SW846 6010B SW846 6020
Antimony	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Arsenic	Aqueous	EPA 200.8	EPA 200.8	SW846 7060A SW846 6010B SW846 6010B Tr SW846 6020	SW846 7060A SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 7060A SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 7060A SW846 6010B SW846 6010B Tr SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Barium	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Beryllium	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Bismuth	Aqueous	200.8	200.8	SW846 6020	-
	Solid	-	-	SW846 6020	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Boron	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6020
Cadmium	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Calcium	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6020
Cerium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Cesium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Chromium	Aqueous	EPA 200.8	EPA 200.8	SW846 7191 SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 7191 SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Cobalt	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Copper	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Dysprosium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW 846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Erbium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Europium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Gallium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Gadolinium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6060	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Germanium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Hafnium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Hardness	Aqueous	-	SM18 2340 C EPA 130.2	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Holmium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Iron	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6020
Iridium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Lanthanum	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Lead	Aqueous	EPA 200.8	EPA 200.8	SW846 7421 SW846 6010B SW846 6010B Tr SW846 6020	SW846 7421 SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 7421 SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 7421 SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 7421 SW846 6010B SW846 6010B Tr SW846 6020
Lithium	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6020
Lutetium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Magnesium	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6020
Manganese	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6020
Mercury	Aqueous	200.8	200.8	SW846 7470A SW846 6020	SW846 7470A SW846 6020
	Solid	-	-	SW846 7471A	-
	Waste	-	-	SW846 7471A	-
	Biological	-	-	SW846 7471A	-
	Air	-	-	-	SW846 7471A SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Molybdenum	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6020
Nickel	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Neodymium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Niobium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Phosphorus	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6020	SW846 6010B SW846 6020
	Air	-	-	-	SW846 6020
Palladium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Potassium	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6020
Platinum	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Praseodymium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Rhenium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Rhodium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Rubidium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Ruthenium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Samarium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Scandium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Selenium	Aqueous	EPA 200.8	EPA 200.8	SW846 7740 SW846 6010B SW846 6010B Tr SW846 6020	SW846 7740 SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 7740 SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 7740 SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Silicon	Aqueous	-	-	SW846 6010B SW846 6010B Tr	-
	Solid	-	-	SW846 6010B	-
	Waste	-	-	SW846 6010B	-
	Biological	-	-	SW846 6010B	-
	Air	-	-	-	-

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Silver	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6010B Tr SW846 6020
Sodium	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6020
Strontium	Aqueous	EPA 200.7	EPA 200.7	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6010B SW846 6020
Sulfur	Aqueous	-	-	SW846 6010B SW846 6010B Tr	SW846 6010B
	Solid	-	-	SW846 6010B	-
	Waste	-	-	SW846 6010B	-
	Biological	-	-	SW846 6010B	-
	Air	-	-	-	SW846 6010B

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Tantalum	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Tellurium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Terbium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Thallium	Aqueous	EPA 200.8	EPA 200.8	SW846 7841 SW846 6010B SW846 6010B Tr SW846 6020	SW846 7841 SW846 6010B SW846 6010B Tr SW846 6020
	Solid	-	-	SW846 7841 SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 7841 SW846 6010B SW846 6010B Tr SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Tin	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6020
Thorium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Thulium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Titanium	Aqueous	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6020
	Solid	-	-	SW846 6010B SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6020
Tungsten	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Uranium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Vanadium	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	-	SW846 6020
Ytterbium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020
Yttrium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-4
SACRAMENTO LABORATORY
Metals Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Zinc	Aqueous	EPA 200.8	EPA 200.8	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
	Solid	-	-	SW846 6010B SW846 6010B Tr SW846 6020	-
	Waste	-	-	SW846 6010B SW846 6020	-
	Biological	-	-	SW846 6010B SW846 6020	-
	Air	-	-	SW846 6010B SW846 6010B Tr SW846 6020	SW846 6010B SW846 6020
Zirconium	Aqueous	-	-	SW846 6020	SW846 6020
	Solid	-	-	SW846 6020	-
	Waste	-	-	SW846 6020	-
	Biological	-	-	SW846 6020	-
	Air	-	-	-	SW846 6020

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Alkalinity (carbonate, bicarbonate, total)	Aqueous	-	EPA 310.1 SM18 2320B	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Ammonia	Aqueous	-	EPA 350.1	-	-
	Solid	-	EPA 350.1	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Adsorbable Organic Halides (AOX)	Aqueous	-	EPA 1650A	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Bromide	Aqueous	-	EPA 300.0	SW846 9056	CARB 421 EPA 26A SW846 9057
	Solid	-	EPA 300.0	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	CARB 421 EPA 26A SW846 9057
Carbon, Total Inorganic	Aqueous	-	EPA 415.1	-	-
	Solid	-	-	SW846 9060	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Carbon, Total Organic	Aqueous	-	EPA 415.1 SM18 5310C	SW846 9060	-
	Solid	-	-	SW846 9060 Walkley-Black	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Chloride	Aqueous	-	EPA 300.0 EPA 325.2 EPA 325.3	SW846 9056 SW846 9251	CARB 421 EPA 26A SW846 9057
	Solid	-	EPA 300.0	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	CARB 421 EPA 26A SW846 9057
Chromium, Hexavalent	Aqueous	-	SM18 3500-D	SW846 7196A	CARB 425 SW846 0061 EPA 306
	Solid	-	-	SW846 7196A	-
	Waste	-	-	SW846 7196A	-
	Biological	-	-	-	-
	Air	-	-	-	CARB 425 SW846 0061 EPA 306
Corrosivity	Aqueous	-	-	-	-
	Solid	-	-	SW846 9040B	-
	Waste	-	-	SW846 9040B	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Conductivity	Aqueous	-	EPA 120.1	SW846 9050A	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Cyanide, Free	Aqueous	-	SM18 4500CN I	SW846 9012A	-
	Solid	-	SM18 4500CN I	SW846 9012A	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Cyanide, Total	Aqueous	-	EPA 335.4	SW846 9012A	CARB 426
	Solid	-	EPA 335.4	SW846 9012A	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	CARB 426
Demand, Chemical Oxygen	Aqueous	-	EPA 410.4	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Fluoride	Aqueous	-	EPA 300.0 EPA 340.2	SW846 9056	CARB 421 EPA 26A SW846 9057
	Solid	-	EPA 300.0 EPA 340.2	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	CARB 421 EPA 26A SW846 9057

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
	Air	-	-	-	CARB 421 EPA 26A SW846 9057
Halogens, Total Organic	Aqueous	-	-	SW846 9020B	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Hardness	Aqueous	-	EPA 130.2 SM18 2340 C	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
N-Hexane Extractable Materials	Aqueous	-	EPA 1664	EPA 1664	-
	Solid	-	EPA 1664	EPA 1664	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Ignitability	Aqueous	-	-	SW846 1020A	-
	Solid	-	-	SW846 1020A	-
	Waste	-	-	SW846 1020A	-
	Biological	-	-	-	-
	Air	-	-	-	-
Moisture	Aqueous	-	-	-	-
	Solid	-	-	ASTM 2216	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Nitrate	Aqueous	-	EPA 300.0 EPA 353.2	SW846 9056	CARB 421
	Solid	-	EPA 300.0	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	CARB 421
Nitrate-Nitrite	Aqueous	-	EPA 353.2	-	-
	Solid	-	EPA 353.2	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Nitrite	Aqueous	-	EPA 300.0 EPA 353.2	SW846 9056	CARB 421
	Solid	-	EPA 300.0	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	CARB 421
Nitrogen, Organic	Aqueous	-	EPA 350.1	-	EPA 350.1
	Solid	-	EPA 350.1	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	EPA 350.1
Nitrogen, Total	Aqueous	-	EPA 351.2 EPA 350.1	-	-
	Solid *	-	EPA 351.2 EPA 350.1	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Nitrogen, Total Kjeldahl	Aqueous	-	EPA 351.2	-	-
	Solid	-	EPA 351.2	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Orthophosphate	Aqueous	-	EPA 300.0 EPA 365.3	SW846 9056	-
	Solid	-	EPA 300.0 EPA 365.3	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Phosphorus, Total	Aqueous	-	EPA 365.3	-	-
	Solid	-	EPA 365.3	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Particulates in Air	Aqueous	-	-	-	EPA 5 EPA PM-10
	Solid	-	-	SW846 9095	-
	Waste	-	-	SW846 9095	-
	Biological	-	-	-	-
	Air	-	-	-	EPA 5 EPA PM-10
pH	Aqueous	-	EPA 150.1	SW846 9041A	-
	Solid	-	-	SW846 9045C	-
	Waste	-	-	SW846 9041A SW846 9045C	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Phenolics	Aqueous	-	EPA 420.4	SW846 9066	-
	Solid	-	EPA 420.4	SW846 9066	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Solids, Total	Aqueous	-	EPA 160.3	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Solids, Total Dissolved	Aqueous	-	EPA 160.1	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Solids, Total Suspended	Aqueous	-	EPA 160.2	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Settleable Solids	Aqueous	-	EPA 160.5	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Sulfate	Aqueous	-	EPA 300.0	SW846 9056	-
	Solid	-	EPA 300.0 EPA 375.4	-	-
	Waste	-	EPA 375.4	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Sulfide	Aqueous	-	EPA 376.2	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-
Nitrocellulose	Aqueous	-	EPA 353.2	-	-
	Solid	-	EPA 353.2	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

TABLE 4-5
SACRAMENTO LABORATORY
Wet Chemistry Methods

Parameter	Matrix	Field of Testing			
		SDWA	CWA	RCRA/CERCLA	CAA
Turbidity	Aqueous	-	EPA 180.1	-	-
	Solid	-	-	-	-
	Waste	-	-	-	-
	Biological	-	-	-	-
	Air	-	-	-	-

Facility Appendix

Sacramento Laboratory

Section Sacramento -5

MDLs and RLs

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TABLE 5-1
SACRAMENTO LABORATORY
GC Volatile CA LUFT Method and BTEX by 8021B
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Benzene	71-43-2	DHS CA LUFT	3/99	0.150 ug/L	1.0 ug/L	12/98	0.232 ug/kg	1.0 ug/kg
Ethylbenzene	100-41-4	DHS CA LUFT	3/99	0.152 ug/L	1.0 ug/L	12/98	0.252 ug/kg	1.0 ug/kg
TPH (as Gasoline)	39-40-0	DHS CA LUFT	1/99	25 ug/L	50 ug/L	1/99	500 ug/kg	1000 ug/kg
Methyl tert-butyl ether	1634-04-4	DHS CA LUFT	4/98	2.5 ug/L	5.0 ug/L	12/98	0.349 ug/kg	5.0 ug/kg
Toluene	108-88-3	DHS CA LUFT	3/99	0.132 ug/L	1.0 ug/L	12/98	1.0 ug/kg	1.0 ug/kg
m-Xylene & p-Xylene	136777-61-2	DHS CA LUFT	3/99	0.284 ug/L	2.0 ug/L	12/98	0.593 ug/kg	2.0 ug/kg
o-Xylene	95-47-6	DHS CA LUFT	3/99	0.132 ug/L	1.0 ug/L	12/98	0.302 ug/kg	1.0 ug/kg

TABLE 5-2
SACRAMENTO LABORATORY
Method 8021B
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Benzene	71-43-2	SW846 8021B	3/99	0.15 ug/L	1.0 ug/L	12/98	0.349 ug/kg	1.0 ug/kg
Bromodichloromethane	75-27-4	SW846 8021B	3/99	0.14 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
Bromoform	75-25-2	SW846 8021B	3/99	0.07 ug/L	5.0 ug/L	12/98	2.5 ug/kg	5.0 ug/kg
Bromomethane	74-83-9	SW846 8021B	3/99	0.16 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
Carbon tetrachloride	56-23-5	SW846 8021B	6/98	0.14 ug/L	1.0 ug/L	6/98	0.5 ug/kg	1.0 ug/kg
Chlorobenzene	108-90-7	SW846 8021B	6/98	0.14 ug/L	1.0 ug/L	6/98	0.285 ug/kg	1.0 ug/kg
Chlorodibromomethane	124-48-1	SW846 8021B	3/99	0.11 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
Chloroethane	75-00-3	SW846 8021B	3/99	0.11 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
2-Chloroethyl vinyl ether	110-75-8	SW846 8021B	3/99	0.11 ug/L	5.0 ug/L	4/98	2.5 ug/kg	5.0 ug/kg
Chloroform	67-66-3	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	6/98	0.5 ug/kg	1.0 ug/kg
Chloromethane	74-87-3	SW846 8021B	3/99	0.24 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
1,2-Dibromoethane	106-93-4	SW846 8021B	3/99	0.10 ug/L	5.0 ug/L	12/98	2.5 ug/kg	5.0 ug/kg
1,2-Dichlorobenzene	95-50-1	SW846 8021B	3/99	0.14 ug/L	1.0 ug/L	12/98	1.0 ug/kg	1.0 ug/kg
1,3-Dichlorobenzene	541-73-1	SW846 8021B	3/99	0.14 ug/L	1.0 ug/L	12/98	0.389 ug/kg	1.0 ug/kg
1,4-Dichlorobenzene	106-46-7	SW846 8021B	3/99	0.14 ug/L	1.0 ug/L	12/98	0.399 ug/kg	1.0 ug/kg
Dichlorodifluoromethane	75-71-8	SW846 8021B	3/99	0.12 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
1,1-Dichloroethane	75-34-3	SW846 8021B	3/99	0.14 ug/L	1.0 ug/L	6/98	0.5 ug/kg	1.0 ug/kg
1,2-Dichloroethane	107-06-2	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
1,1-Dichloroethene	75-35-4	SW846 8021B	3/99	0.12 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
cis-1,2-Dichloroethene	156-59-2	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
trans-1,2-Dichloroethene	156-60-5	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
1,2-Dichloroethene, Total	540-59-0	SW846 8021B	3/99	-- ug/L	ug/L	1/99	-- ug/kg	ug/kg
1,2-Dichloropropane	78-87-5	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
cis-1,3-Dichloropropene	10061-01-5	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
trans-1,3-Dichloropropene	10061-02-6	SW846 8021B	3/99	0.12 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
Ethylbenzene	100-41-4	SW846 8021B	3/99	0.15 ug/L	1.0 ug/L	12/98	0.252 ug/kg	1.0 ug/kg
Methylene Chloride	75-09-2	SW846 8021B	3/99	0.15 ug/L	5.0 ug/L	6/98	2.5 ug/kg	5.0 ug/kg
Methyl tert-butyl ether	1634-04-4	SW846 8021B	3/99	0.5 ug/L	1.0 ug/L	1/99	0.349 ug/kg	1.0 ug/kg
1,1,2,2-Tetrachloroethane	79-34-5	SW846 8021B	3/99	0.31 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
Tetrachloroethene	127-18-4	SW846 8021B	3/99	0.15 ug/L	1.0 ug/L	6/98	0.5 ug/kg	1.0 ug/kg
Toluene	108-88-3	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	12/98	0.375 ug/kg	1.0 ug/kg
1,1,1-Trichloroethane	71-55-6	SW846 8021B	3/99	0.18 ug/L	1.0 ug/L	6/98	0.5 ug/kg	1.0 ug/kg
1,1,2-Trichloroethane	79-00-5	SW846 8021B	6/98	0.16 ug/L	1.0 ug/L	6/98	0.5 ug/kg	1.0 ug/kg
Trichloroethene	79-01-6	SW846 8021B	3/99	0.18 ug/L	1.0 ug/L	6/98	0.5 ug/kg	1.0 ug/kg
Trichlorofluoromethane	75-69-4	SW846 8021B	3/99	0.11 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	SW846 8021B	3/99	0.13 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
Vinyl chloride	75-01-4	SW846 8021B	3/99	0.33 ug/L	1.0 ug/L	12/98	0.5 ug/kg	1.0 ug/kg
m-Xylene & p-Xylene	136777-61-2	SW846 8021B	3/99	0.28 ug/L	2.0 ug/L	12/98	0.593 ug/kg	2.0 ug/kg
o-Xylene	95-47-6	SW846 8021B	3/99	0.14 ug/L	1.0 ug/L	12/98	0.302 ug/kg	1.0 ug/kg

TABLE 5-3
SACRAMENTO LABORATORY
Method 504.1
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
3-Chloro-1,2-dibromopropane	96-12-8	EPA-DW 504.1	6/99	0.02 ug/L	0.05 ug/L		NR	NR
1,2-Dibromoethane	106-93-4	EPA-DW 504.1	6/99	0.02 ug/L	0.05 ug/L		NR	NR

TABLE 5-4
SACRAMENTO LABORATORY
GC Semi-Volatile Method 8015M
Method Detection Limits (MDL) and
Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
TPH (as Diesel)	Q797	SW846 8015 MOD	1/99	14 ug/L	50 ug/L	5/99	0.57 mg/kg	1.0 mg/kg
TPH (as Motor Oil)	Q1770	SW846 8015 MOD	2/98	28.ug/L	250 ug/L	8/97	1.56 mg/kg	5.0 mg/kg

TABLE 5-5
SACRAMENTO LABORATORY
Method 8081A
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Aldrin	309-00-2	SW846 8081A	5/98	0.006 ug/L	0.05 ug/L	12/98	0.36 ug/kg	1.7 ug/kg
alpha-BHC	319-84-6	SW846 8081A	5/98	0.008 ug/L	0.05 ug/L	12/98	0.39 ug/kg	1.7 ug/kg
beta-BHC	319-85-7	SW846 8081A	5/98	0.004 ug/L	0.05 ug/L	12/98	0.38 ug/kg	1.7 ug/kg
delta-BHC	319-86-8	SW846 8081A	5/98	0.006 ug/L	0.05 ug/L	12/98	0.44 ug/kg	1.7 ug/kg
gamma-BHC (Lindane)	58-89-9	SW846 8081A	5/98	0.01 ug/L	0.05 ug/L	12/98	0.35 ug/kg	1.7 ug/kg
alpha-Chlordane	5103-71-9	SW846 8081A	5/98	0.007 ug/L	0.05 ug/L	12/98	0.31 ug/kg	1.7 ug/kg
gamma-Chlordane	5103-74-2	SW846 8081A	5/98	0.008 ug/L	0.05 ug/L	12/98	0.32 ug/kg	1.7 ug/kg
Chlordane(technical)	57-74-9	SW846 8081A	5/98	2.5 ug/L	5.0 ug/L	12/98	8.5 ug/kg	17 ug/kg
p-Chlorobenzilate	510-15-6	SW846 8081A	5/98	0.5 ug/L	1.0 ug/L	12/98	17.5 ug/kg	35 ug/kg
4,4'-DDD	72-54-8	SW846 8081A	5/98	0.005 ug/L	0.10 ug/L	12/98	0.71 ug/kg	3.4 ug/kg
2,4'-DDD	53-19-0	SW846 8081A	5/99	0.05 ug/L	0.10 ug/L	5/99	1.7 ug/kg	3.4 ug/kg
4,4'-DDE	72-55-9	SW846 8081A	5/98	0.015 ug/L	0.10 ug/L	12/98	0.63 ug/kg	3.4 ug/kg
2,4'-DDE	3424-82-6	SW846 8081A	5/99	0.05 ug/L	0.10 ug/L	5/99	1.7 ug/kg	3.4 ug/kg
4,4'-DDT	50-29-3	SW846 8081A	5/98	0.02 ug/L	0.10 ug/L	12/98	0.31 ug/kg	1.7 ug/kg
2,4'-DDT	789-02-6	SW846 8081A	5/99	0.05 ug/L	0.10 ug/L	5/99	1.7 ug/kg	3.4 ug/kg
[illegible]late	2303-16-4	SW846 8081A	5/98	0.5 ug/L	1.0 ug/L	12/98	17.5 ug/kg	35 ug/kg
[illegible]drin	60-57-1	SW846 8081A	5/98	0.02 ug/L	0.10 ug/L	12/98	0.51 ug/kg	3.4 ug/kg
Endosulfan I	959-98-8	SW846 8081A	5/98	0.010 ug/L	0.05 ug/L	12/98	0.38 ug/kg	1.7 ug/kg
Endosulfan II	33213-65-9	SW846 8081A	5/98	0.02 ug/L	0.10 ug/L	12/98	0.72 ug/kg	3.4 ug/kg
Endosulfan sulfate	1031-07-8	SW846 8081A	5/98	0.033 ug/L	0.10 ug/L	12/98	0.82 ug/kg	3.4 ug/kg
Endrin	72-20-8	SW846 8081A	5/98	0.02 ug/L	0.10 ug/L	12/98	0.49 ug/kg	3.4 ug/kg
Endrin aldehyde	7421-93-4	SW846 8081A	5/98	0.029 ug/L	0.10 ug/L	12/98	1.16 ug/kg	3.4 ug/kg
Endrin ketone	53494-70-5	SW846 8081A	5/98	0.024 ug/L	0.10 ug/L	12/98	0.79 ug/kg	3.4 ug/kg
Heptachlor	76-44-8	SW846 8081A	5/98	0.007 ug/L	0.05 ug/L	12/98	0.38 ug/kg	1.7 ug/kg
Heptachlor epoxide	1024-57-3	SW846 8081A	5/98	0.008 ug/L	0.05 ug/L	12/98	0.25 ug/kg	1.7 ug/kg
Isodrin	465-73-6	SW846 8081A	5/98	0.5 ug/L	1.0 ug/L	12/98	17.5 ug/kg	35 ug/kg
Methoxychlor	72-43-5	SW846 8081A	5/98	0.04 ug/L	2.0 ug/L	12/98	2.6 ug/kg	17 ug/kg
Mirex	2385-85-5	SW846 8081A	2/98	0.05 ug/L	0.1 ug/L	5/98	0.85 ug/kg	1.7 ug/kg
Toxaphene	8001-35-2	SW846 8081A	5/98	1.4 ug/L	2.0 ug/L	12/98	56 ug/kg	67 ug/kg

TABLE 5-6
SACRAMENTO LABORATORY
Method 8082
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
PCB-1016	12674-11-2	SW846 8082	5/98	0.21 ug/L	1.0 ug/L	5/98	1.9 ug/kg	33 ug/kg
PCB-1221	11104-28-2	SW846 8082	5/98	0.37 ug/L	1.0 ug/L	5/98	1.9 ug/kg	33 ug/kg
PCB-1232	11141-16-5	SW846 8082	5/98	0.37 ug/L	1.0 ug/L	5/98	1.9 ug/kg	33 ug/kg
PCB-1242	53469-21-9	SW846 8082	5/98	0.37 ug/L	1.0 ug/L	5/98	1.9 ug/kg	33 ug/kg
PCB-1248	12672-29-6	SW846 8082	5/98	0.37 ug/L	1.0 ug/L	5/98	1.9 ug/kg	33 ug/kg
PCB-1254	11097-69-1	SW846 8082	5/98	0.37 ug/L	1.0 ug/L	5/98	1.9 ug/kg	33 ug/kg
PCB-1260	11096-82-5	SW846 8082	5/98	0.37 ug/L	1.0 ug/L	5/98	1.7 ug/kg	33 ug/kg

TABLE 5-7
SACRAMENTO LABORATORY
Method 8151A
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
2,4-D	94-75-7	SW846 8151A	8/98	0.515 ug/L	4.0 ug/L	8/98	40 ug/kg	80 ug/kg
Dalapon	75-99-0	SW846 8151A	8/98	0.196 ug/L	2.0 ug/L	8/98	20 ug/kg	40 ug/kg
2,4-DB	94-82-6	SW846 8151A	8/98	0.488 ug/L	4.0 ug/L	8/98	40 ug/kg	80 ug/kg
Dicamba	1918-00-9	SW846 8151A	8/98	0.268 ug/L	2.0 ug/L	8/98	20 ug/kg	40 ug/kg
Dichlorprop	120-36-5	SW846 8151A	8/98	0.512 ug/L	4.0 ug/L	8/98	40 ug/kg	80 ug/kg
Dinoseb	88-85-7	SW846 8151A	8/98	0.431 ug/L	0.60 ug/L	8/98	6.0 ug/kg	12 ug/kg
MCPA	94-74-6	SW846 8151A	12/98	200 ug/L	400 ug/L	4/98	4000 ug/kg	8000 ug/kg
MCPP	7085-19-0	SW846 8151A	12/98	200 ug/L	400 ug/L	4/98	4000 ug/kg	8000 ug/kg
Pentachlorophenol	87-86-5	SW846 8151A	12/98	0.25 ug/L	0.50 ug/L	4/98	0.25 ug/kg	2.5 ug/L
Silvex (2,4,5-TP)	93-72-1	SW846 8151A	8/98	0.136 ug/L	1.0 ug/L	4/98	10 ug/kg	5.0 ug/L
2,4,5-T	93-76-5	SW846 8151A	8/98	0.134 ug/L	1.0 ug/L	8/98	10 ug/kg	5.0 ug/L
2,3,4,6-Tetrachlorophenol	58-90-2	SW846 8151A	8/98	0.134 ug/L	0.1 ug/L	8/98	0.25 ug/kg	2.5 ug/L

TABLE 5-8
SACRAMENTO LABORATORY
Method 8310
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Acenaphthene	83-32-9	SW846 8310	12/97	0.26 ug/L	2.0 ug/L	7/98	63 ug/kg	400 ug/kg
Acenaphthylene	208-96-8	SW846 8310	12/97	0.076 ug/L	1.0 ug/L	7/98	46 ug/kg	200 ug/kg
Anthracene	120-12-7	SW846 8310	12/97	0.009 ug/L	0.04 ug/L	7/98	1.1 ug/kg	8 ug/kg
Benzo[a]anthracene	56-55-3	SW846 8310	12/97	0.005 ug/L	0.08 ug/L	7/98	1.7 ug/kg	16 ug/kg
Benzo[b]fluoranthene	205-99-2	SW846 8310	12/97	0.006 ug/L	0.02 ug/L	4/98	2.4 ug/kg	4.0 ug/kg
Benzo[k]fluoranthene	207-08-9	SW846 8310	12/97	0.013 ug/L	0.02 ug/L	7/98	1.1 ug/kg	4.0 ug/kg
Benzo[g,h,i]perylene	191-24-2	SW846 8310	12/97	0.016 ug/L	0.08 ug/L	7/98	3.1 ug/kg	16 ug/kg
Benzo[a]pyrene	50-32-8	SW846 8310	12/97	0.006 ug/L	0.05 ug/L	7/98	3.1 ug/kg	10 ug/kg
Chrysene	218-01-9	SW846 8310	12/97	0.002 ug/L	0.1 ug/L	7/98	14 ug/kg	20 ug/kg
Dibenz(a,h)anthracene	53-70-3	SW846 8310	12/97	0.018 ug/L	0.2 ug/L	7/98	9.2 ug/kg	40 ug/kg
Fluoranthene	206-44-0	SW846 8310	12/97	0.022 ug/L	0.1 ug/L	7/98	4.8 ug/kg	20 ug/kg
Fluorene	86-73-7	SW846 8310	12/97	0.029 ug/L	0.2 ug/L	7/98	6.7 ug/kg	40 ug/kg
Indeno[1,2,3-cd]pyrene	193-39-5	SW846 8310	12/97	0.0096 ug/L	0.10 ug/L	7/98	3.1 ug/kg	20 ug/kg
Naphthalene	91-20-3	SW846 8310	5/97	0.57 ug/L	1.0 ug/L	7/98	23 ug/kg	200 ug/kg
Phenanthrene	85-01-8	SW846 8310	12/97	0.024 ug/L	0.08 ug/L	7/98	2.6 ug/kg	16 ug/kg
Pyrene	129-00-0	SW846 8310	12/97	0.043 ug/L	0.2 ug/L	7/98	11 ug/kg	40 ug/kg

TABLE 5-9
SACRAMENTO LABORATORY
Method 8330
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
2-Amino-4,6-dinitrotoluene	35572-78-2	SW846 8330	7/98	5.0ug/L	10 ug/L	7/98	0.065 mg/kg	0.25 mg/kg
4-Amino-2,6-dinitrotoluene	19406-51-0	SW846 8330	7/98	4.4 ug/L	10 ug/L	7/98	0.098 mg/kg	0.25 mg/kg
3,5-Dinitroaniline	618-87-1	SW846 8330	5/99	1.86 ug/L	25 ug/L	5/99	0.057 mg/kg	0.5 mg/kg
1,3-Dinitrobenzene	99-65-0	SW846 8330	7/98	0.74 ug/L	4.0 ug/L	7/98	0.017 mg/kg	0.25 mg/kg
2,4-Dinitrotoluene	121-14-2	SW846 8330	5/99	1.9 ug/L	5.7 ug/L	7/98	0.028 mg/kg	0.25 mg/kg
2,6-Dinitrotoluene	606-20-2	SW846 8330	7/98	3.0ug/L	9.4 ug/L	7/98	0.070 mg/kg	0.25 mg/kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4	SW846 8330	7/98	7.0 ug/L	14.0 ug/L	7/98	0.051 mg/kg	0.25 mg/kg
Methyl-2,4,6-trinitrophenylnitramine	479-45-8	SW846 8330	7/98	2.0 ug/L	4.0 ug/L	7/98	0.025 mg/kg	0.25 mg/kg
Nitrobenzene	98-95-3	SW846 8330	7/98	1.8 ug/L	6.4 ug/L	7/98	0.025 mg/kg	0.25 mg/kg
Nitroglycerin	55-63-0	SW846 8330	5/99	1.87 ug/L	25 ug/L	2/99	0.5 mg/kg	1.0 mg/kg
m-Nitrotoluene	99-08-1	SW846 8330	7/98	3.6 ug/L	7.9 ug/L	7/98	0.125 mg/kg	0.25 mg/kg
p-Nitrotoluene	99-99-0	SW846 8330	7/98	4.25 ug/L	8.5 ug/L	7/98	0.125 mg/kg	0.25 mg/kg
o-Nitrotoluene	88-72-2	SW846 8330	7/98	6.0 ug/L	12.0 ug/L	7/98	0.014 mg/kg	0.25 mg/kg
[illegible]hydro-1,3,5,7-tetranitro-[illegible]5,7-tetrazocine	2691-41-0	SW846 8330	7/98	0.5 ug/L	13.0 ug/L	7/98	0.014 mg/kg	0.25 mg/kg
Pentaerythritol Tetranitrate	78-11-5	SW846 8330	2/99	4.92 ug/L	50 ug/L	2/99	0.5 mg/kg	0.5 mg/kg
1,3,5-Trinitrobenzene	99-35-4	SW846 8330	7/98	2.0 ug/L	7.3 ug/L	7/98	0.015 mg/kg	0.25 mg/kg
2,4,6-Trinitrotoluene	118-96-7	SW846 8330	7/98	1.6 ug/L	6.9 ug/L	7/98	0.031 mg/kg	0.25 mg/kg

TABLE 5-10
SACRAMENTO LABORATORY
Method 8260B
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Acetone	67-64-1	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Acetonitrile	75-05-8	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Acrolein	107-02-8	SW846 8260B	5/98	10 ug/L	20 ug/L	7/98	50 ug/kg	100 ug/kg
Acrylonitrile	107-13-1	SW846 8260B	5/98	10 ug/L	20 ug/L	7/98	50 ug/kg	100 ug/kg
Benzene	71-43-2	SW846 8260B	5/98	0.13 ug/L	1.0 ug/L	7/98	0.73 ug/kg	5.0 ug/kg
Bromobenzene	108-86-1	SW846 8260B	5/98	0.18 ug/L	1.0 ug/L	7/98	0.52 ug/kg	5.0 ug/kg
Bromochloromethane	74-97-5	SW846 8260B	5/98	0.31 ug/L	1.0 ug/L	7/98	0.94 ug/kg	5.0 ug/kg
Bromodichloromethane	75-27-4	SW846 8260B	5/98	0.14 ug/L	1.0 ug/L	7/98	0.53 ug/kg	5.0 ug/kg
Bromoform	75-25-2	SW846 8260B	5/98	0.10 ug/L	1.0 ug/L	7/98	4.1 ug/kg	5.0 ug/kg
Bromomethane	74-83-9	SW846 8260B	5/98	0.08 ug/L	1.0 ug/L	7/98	0.86 ug/kg	5.0 ug/kg
2-Butanone	78-93-3	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
n-Butylbenzene	104-51-8	SW846 8260B	5/98	0.12 ug/L	1.0 ug/L	7/98	0.66 ug/kg	5.0 ug/kg
sec-Butylbenzene	135-98-8	SW846 8260B	5/98	0.12 ug/L	1.0 ug/L	7/98	0.75 ug/kg	5.0 ug/kg
tert-Butylbenzene	98-06-6	SW846 8260B	5/98	0.14 ug/L	1.0 ug/L	7/98	0.54 ug/kg	5.0 ug/kg
Carbon disulfide	75-15-0	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Carbon tetrachloride	56-23-5	SW846 8260B	5/98	0.15 ug/L	1.0 ug/L	7/98	0.53 ug/kg	5.0 ug/kg
Chlorobenzene	108-90-7	SW846 8260B	5/98	0.12 ug/L	1.0 ug/L	7/98	0.79 ug/kg	5.0 ug/kg
2-Chloro-1,3-butadiene	126-99-8	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Chlorodibromomethane	124-48-1	SW846 8260B	5/98	0.40 ug/L	1.0 ug/L	7/98	2.7 ug/kg	5.0 ug/kg
Chloroethane	75-00-3	SW846 8260B	5/98	0.34 ug/L	1.0 ug/L	7/98	2.6 ug/kg	5.0 ug/kg
2-Chloroethyl vinyl ether	110-75-8	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
Chloroform	67-66-3	SW846 8260B	5/98	0.72 ug/L	1.0 ug/L	7/98	0.75 ug/kg	5.0 ug/kg
1-Chlorohexane	544-10-5	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
Chloromethane	74-87-3	SW846 8260B	5/98	0.25 ug/L	1.0 ug/L	7/98	1.5 ug/kg	5.0 ug/kg
3-Chloro-1-propene	107-05-1	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	2.5 ug/kg	5.0 ug/kg
2-Chlorotoluene	95-49-8	SW846 8260B	5/98	0.26 ug/L	1.0 ug/L	7/98	0.62 ug/kg	5.0 ug/kg
4-Chlorotoluene	106-43-4	SW846 8260B	5/98	0.10 ug/L	1.0 ug/L	7/98	0.86 ug/kg	5.0 ug/kg
3-Chloro-1,2-dibromopropane	96-12-8	SW846 8260B	5/98	0.95 ug/L	2.0 ug/L	7/98	7.2 ug/kg	10 ug/kg
1,2-Dibromoethane	106-93-4	SW846 8260B	5/98	0.22 ug/L	2.0 ug/L	7/98	0.79 ug/kg	10 ug/kg
Dibromomethane	74-95-3	SW846 8260B	5/98	0.21 ug/L	1.0 ug/L	7/98	0.58 ug/kg	5.0 ug/kg
1,2-Dichlorobenzene	95-50-1	SW846 8260B	5/98	0.14 ug/L	1.0 ug/L	7/98	0.64 ug/kg	5.0 ug/kg
1,3-Dichlorobenzene	541-73-1	SW846 8260B	5/98	0.11 ug/L	1.0 ug/L	7/98	0.75 ug/kg	5.0 ug/kg
1,4-Dichlorobenzene	106-46-7	SW846 8260B	5/98	0.13 ug/L	1.0 ug/L	7/98	0.78 ug/kg	5.0 ug/kg
trans-1,4-Dichloro-2-butene	110-57-6	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Dichlorodifluoromethane	75-71-8	SW846 8260B	5/98	0.16 ug/L	1.0 ug/L	7/98	0.89 ug/kg	5.0 ug/kg
1,1-Dichloroethane	75-34-3	SW846 8260B	5/98	0.10 ug/L	1.0 ug/L	7/98	0.76 ug/kg	5.0 ug/kg
1,2-Dichloroethane	107-06-2	SW846 8260B	5/98	0.22 ug/L	1.0 ug/L	7/98	0.73 ug/kg	5.0 ug/kg
1,1-Dichloroethene	75-35-4	SW846 8260B	5/98	0.36 ug/L	1.0 ug/L	7/98	1.2 ug/kg	5.0 ug/kg
cis-1,2-Dichloroethene	156-59-2	SW846 8260B	5/98	0.10 ug/L	1.0 ug/L	7/98	0.89 ug/kg	5.0 ug/kg
trans-1,2-Dichloroethene	156-60-5	SW846 8260B	5/98	0.11 ug/L	1.0 ug/L	7/98	0.91 ug/kg	5.0 ug/kg
1,2-Dichloroethene, Total	540-59-0	SW846 8260B	5/98	0.0 ug/L	1.0 ug/L	7/98	0.0 ug/kg	5.0 ug/kg
1,2-Dichloropropane	78-87-5	SW846 8260B	5/98	0.15 ug/L	1.0 ug/L	7/98	0.60 ug/kg	5.0 ug/kg
1,3-Dichloropropane	142-28-9	SW846 8260B	5/98	0.20 ug/L	1.0 ug/L	7/98	0.57 ug/kg	5.0 ug/kg
2,2-Dichloropropane	594-20-7	SW846 8260B	5/98	0.13 ug/L	1.0 ug/L	7/98	1.1 ug/kg	5.0 ug/kg

TABLE 5-10
SACRAMENTO LABORATORY
Method 8260B
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
1,1-Dichloropropene	563-58-6	SW846 8260B	5/98	0.14 ug/L	1.0 ug/L	7/98	0.86 ug/kg	5.0 ug/kg
cis-1,3-Dichloropropene	10061-01-5	SW846 8260B	5/98	0.22 ug/L	1.0 ug/L	7/98	0.64 ug/kg	5.0 ug/kg
trans-1,3-Dichloropropene	10061-02-6	SW846 8260B	5/98	0.30 ug/L	1.0 ug/L	7/98	0.75 ug/kg	5.0 ug/kg
1,4-Dioxane	123-91-1	SW846 8260B	5/98	25 ug/L	50 ug/L	7/98	125 ug/kg	250 ug/kg
Tert-amyl methyl ether	994-05-8	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
Tert-butyl ethyl ether	637-92-3	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
Ethylbenzene	100-41-4	SW846 8260B	5/98	0.27 ug/L	1.0 ug/L	7/98	0.86 ug/kg	5.0 ug/kg
Ethyl methacrylate	97-63-2	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Hexachlorobutadiene	87-68-3	SW846 8260B	5/98	0.22 ug/L	1.0 ug/L	7/98	0.89 ug/kg	5.0 ug/kg
n-Hexane	110-54-3	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
2-Hexanone	591-78-6	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Iodomethane	74-88-4	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Isobutanol	78-83-1	SW846 8260B	5/98	10 ug/L	20 ug/L	7/98	50 ug/kg	100 ug/kg
Isopropylbenzene	98-82-8	SW846 8260B	5/98	0.12 ug/L	1.0 ug/L	7/98	0.52 ug/kg	5.0 ug/kg
Isopropyl ether	108-20-3	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
4-Isopropyltoluene	99-87-6	SW846 8260B	5/98	0.13 ug/L	1.0 ug/L	7/98	0.63 ug/kg	5.0 ug/kg
Methacrylonitrile	126-98-7	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Methylene Chloride	75-09-2	SW846 8260B	5/98	0.35 ug/L	1.0 ug/L	7/98	0.84 ug/kg	10 ug/kg
Methyl methacrylate	80-62-6	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
4-Methyl-2-pentanone	108-10-1	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
2-Methyl-2-propanol	75-65-0	SW846 8260B	1/99	25 ug/L	50 ug/L	1/99	125 ug/kg	250 ug/kg
Methyl tert-butyl ether	1634-04-4	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
Naphthalene	91-20-3	SW846 8260B	5/98	0.15 ug/L	1.0 ug/L	7/98	0.63 ug/kg	5.0 ug/kg
Propionitrile	107-12-0	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
N-Propylbenzene	103-65-1	SW846 8260B	5/98	0.15 ug/L	1.0 ug/L	7/98	0.90 ug/kg	5.0 ug/kg
Styrene	100-42-5	SW846 8260B	5/98	0.15 ug/L	1.0 ug/L	7/98	0.76 ug/kg	5.0 ug/kg
1,1,1,2-Tetrachloroethane	630-20-6	SW846 8260B	5/98	0.10 ug/L	1.0 ug/L	7/98	3.0 ug/kg	5.0 ug/kg
1,1,2,2-Tetrachloroethane	79-34-5	SW846 8260B	5/98	0.37 ug/L	1.0 ug/L	7/98	0.68 ug/kg	5.0 ug/kg
Tetrachloroethene	127-18-4	SW846 8260B	5/98	0.38 ug/L	1.0 ug/L	7/98	0.61 ug/kg	5.0 ug/kg
Toluene	108-88-3	SW846 8260B	5/98	0.25 ug/L	1.0 ug/L	7/98	0.61 ug/kg	5.0 ug/kg
1,2,3-Trichlorobenzene	87-61-6	SW846 8260B	5/98	0.14 ug/L	1.0 ug/L	7/98	0.75 ug/kg	5.0 ug/kg
1,2,4-Trichlorobenzene	120-82-1	SW846 8260B	5/98	0.23 ug/L	1.0 ug/L	7/98	0.75 ug/kg	5.0 ug/kg
1,1,1-Trichloroethane	71-55-6	SW846 8260B	5/98	0.41 ug/L	1.0 ug/L	7/98	0.80 ug/kg	5.0 ug/kg
1,1,2-Trichloroethane	79-00-5	SW846 8260B	5/98	0.31 ug/L	1.0 ug/L	7/98	2.9 ug/kg	5.0 ug/kg
Trichloroethene	79-01-6	SW846 8260B	5/98	0.31 ug/L	1.0 ug/L	7/98	0.60 ug/kg	5.0 ug/kg
Trichlorofluoromethane	75-69-4	SW846 8260B	5/98	0.23 ug/L	1.0 ug/L	7/98	0.80 ug/kg	5.0 ug/kg
1,2,3-Trichloropropane	96-18-4	SW846 8260B	5/98	0.30 ug/L	1.0 ug/L	7/98	0.76 ug/kg	5.0 ug/kg
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	SW846 8260B	1/99	1.0 ug/L	2.0 ug/L	1/99	5.0 ug/kg	10 ug/kg
1,2,4-Trimethylbenzene	95-63-6	SW846 8260B	5/98	0.12 ug/L	1.0 ug/L	7/98	0.51 ug/kg	5.0 ug/kg
1,3,5-Trimethylbenzene	108-67-8	SW846 8260B	5/98	0.14 ug/L	1.0 ug/L	7/98	2.4 ug/kg	5.0 ug/kg
Vinyl acetate	108-05-4	SW846 8260B	5/98	1.0 ug/L	2.0 ug/L	7/98	5.0 ug/kg	10 ug/kg
Vinyl chloride	75-01-4	SW846 8260B	5/98	0.12 ug/L	1.0 ug/L	7/98	1.6 ug/kg	5.0 ug/kg
m-Xylene & p-Xylene	136777-61-2	SW846 8260B	5/98	0.18 ug/L	1.0 ug/L	7/98	0.81 ug/kg	5.0 ug/kg
o-Xylene	95-47-6	SW846 8260B	5/98	0.10 ug/L	1.0 ug/L	7/98	2.7 ug/kg	5.0 ug/kg

TABLE 5-11
SACRAMENTO LABORATORY
Method 8270C
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Acenaphthene	83-32-9	SW846 8270C	9/98	1.0 ug/L	10 ug/L	2/98	41 ug/kg	330 ug/kg
Acenaphthylene	208-96-8	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	30 ug/kg	330 ug/kg
Acetophenone	98-86-2	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
2-Acetylaminofluorene	53-96-3	SW846 8270C	9/98	50 ug/L	100 ug/L	2/98	1650 ug/kg	3300 ug/kg
4-Aminobiphenyl	92-67-1	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
Aniline	62-53-3	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Anthracene	120-12-7	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	54 ug/kg	330 ug/kg
Aramite, Total	140-57-8	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Azobenzene	103-33-3	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Benzaldehyde	100-52-7	SW846 8270C	3/99	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Benzidine	92-87-5	SW846 8270C	9/98	50 ug/L	100 ug/L	2/98	1650 ug/kg	3300 ug/kg
Benzo[a]anthracene	56-55-3	SW846 8270C	9/98	0.88 ug/L	10 ug/L	2/98	26 ug/kg	330 ug/kg
Benzo[b]fluoranthene	205-99-2	SW846 8270C	9/98	0.87 ug/L	10 ug/L	2/98	48 ug/kg	330 ug/kg
Benzo[j]fluoranthene	205-82-3	SW846 8270C	3/99	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Benzo[k]fluoranthene	207-08-9	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	57 ug/kg	330 ug/kg
Benzoic acid	65-85-0	SW846 8270C	9/98	10 ug/L	50 ug/L	2/98	250 ug/kg	1600 ug/kg
Benzo[g,h,i]perylene	191-24-2	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	56 ug/kg	330 ug/kg
Benzo[a]pyrene	50-32-8	SW846 8270C	9/98	0.83 ug/L	10 ug/L	2/98	40 ug/kg	330 ug/kg
Benzo[e]pyrene	192-97-2	SW846 8270C	3/99	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Benzyl alcohol	100-51-6	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	167 ug/kg	330 ug/kg
1,1'-Biphenyl	92-52-4	SW846 8270C	3/99	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Bis(2-chloroethoxy)methane	111-91-1	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	30 ug/kg	330 ug/kg
bis(2-chloroethyl)ether	111-44-4	SW846 8270C	9/98	1.2 ug/L	10 ug/L	2/98	33 ug/kg	330 ug/kg
bis(2-chloroisopropyl)ether	108-60-1	SW846 8270C	9/98	0.87 ug/L	10 ug/L	2/98	28 ug/kg	330 ug/kg
bis(2-chloroisopropyl)ether	108-60-1	SW846 8270C	9/98	0.87 ug/L	10 ug/L	2/98	28 ug/kg	330 ug/kg
bis(2-ethylhexyl)phthalate	117-81-7	SW846 8270C	9/98	0.71 ug/L	10 ug/L	2/98	43 ug/kg	330 ug/kg
4-Bromophenyl phenyl ether	101-55-3	SW846 8270C	9/98	0.90 ug/L	10 ug/L	2/98	47 ug/kg	330 ug/kg
Butyl benzyl phthalate	85-68-7	SW846 8270C	9/98	0.79 ug/L	10 ug/L	2/98	27 ug/kg	330 ug/kg
Carbazole	86-74-8	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
4-Chloroaniline	106-47-8	SW846 8270C	9/98	0.73 ug/L	10 ug/L	2/98	167 ug/kg	330 ug/kg
p-Chlorobenzilate	510-15-6	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
4-Chloro-3-methylphenol	59-50-7	SW846 8270C	9/98	1.9 ug/L	10 ug/L	2/98	85 ug/kg	330 ug/kg
1-Chloronaphthalene	90-13-1	SW846 8270C	3/99	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
2-Chloronaphthalene	91-58-7	SW846 8270C	9/98	0.97 ug/L	10 ug/L	2/98	20 ug/kg	330 ug/kg
2-Chlorophenol	95-57-8	SW846 8270C	9/98	2.0 ug/L	10 ug/L	2/98	52 ug/kg	330 ug/kg
4-Chlorophenyl phenyl ether	7005-72-3	SW846 8270C	9/98	0.86 ug/L	10 ug/L	2/98	36 ug/kg	330 ug/kg
Chrysene	218-01-9	SW846 8270C	9/98	0.95 ug/L	10 ug/L	2/98	67 ug/kg	330 ug/kg
Diallate	2303-16-4	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Dibenz[a,j]acridine	224-42-0	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Dibenz(a,h)anthracene	53-70-3	SW846 8270C	9/98	0.95 ug/L	10 ug/L	2/98	52 ug/kg	330 ug/kg
Dibenzofuran	132-64-9	SW846 8270C	9/98	1.2 ug/L	10 ug/L	2/98	56 ug/kg	330 ug/kg
Di-n-butyl phthalate	84-74-2	SW846 8270C	9/98	0.97 ug/L	10 ug/L	2/98	62 ug/kg	330 ug/kg
1,2-Dichlorobenzene	95-50-1	SW846 8270C	9/98	0.77 ug/L	10 ug/L	2/98	167 ug/kg	330 ug/kg
1,3-Dichlorobenzene	541-73-1	SW846 8270C	9/98	0.76 ug/L	10 ug/L	2/98	167 ug/kg	330 ug/kg

TABLE 5-11
SACRAMENTO LABORATORY
Method 8270C
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
1,4-Dichlorobenzene	106-46-7	SW846 8270C	9/98	0.75 ug/L	10 ug/L	2/98	167 ug/kg	330 ug/kg
3,3'-Dichlorobenzidine	91-94-1	SW846 8270C	9/98	1.3 ug/L	50 ug/L	2/98	167 ug/kg	1600 ug/kg
2,4-Dichlorophenol	120-83-2	SW846 8270C	9/98	2.0 ug/L	10 ug/L	2/98	157 ug/kg	330 ug/kg
2,6-Dichlorophenol	87-65-0	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Diethyl phthalate	84-66-2	SW846 8270C	9/98	1.0 ug/L	10 ug/L	2/98	41 ug/kg	330 ug/kg
O,O-Diethyl-O-2-pyrazinyl phosphorothioate	297-97-2	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
Dimethoate	60-51-5	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
p-Dimethylamino azobenzene	60-11-7	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
7,12-Dimethylbenz(a)anthracene	57-97-6	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
3,3'-Dimethylbenzidine	119-93-7	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
alpha,alpha-Dimethyl phenethylamine	122-09-8	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
2,4-Dimethylphenol	105-67-9	SW846 8270C	9/98	2.5 ug/L	10 ug/L	2/98	86 ug/kg	330 ug/kg
Dimethyl phthalate	131-11-3	SW846 8270C	9/98	0.96 ug/L	10 ug/L	2/98	25 ug/kg	330 ug/kg
1,3-Dinitrobenzene	99-65-0	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
1,4-Dinitrobenzene	100-25-4	SW846 8270C	3/99	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
4,6-Dinitro-2-methylphenol	534-52-1	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	35 ug/kg	1600 ug/kg
2,4-Dinitrophenol	51-28-5	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	250 ug/kg	1600 ug/kg
2,4-Dinitrotoluene	121-14-2	SW846 8270C	9/98	5.0 ug/L	10 ug/L	2/98	49 ug/kg	330 ug/kg
2,6-Dinitrotoluene	606-20-2	SW846 8270C	9/98	0.76 ug/L	10 ug/L	2/98	167 ug/kg	330 ug/kg
Dinoseb	88-85-7	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Di-n-octyl phthalate	117-84-0	SW846 8270C	9/98	0.84 ug/L	10 ug/L	2/98	41 ug/kg	330 ug/kg
Disulfoton	298-04-4	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
Ethyl methanesulfonate	62-50-0	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Famphur	52-85-7	SW846 8270C	9/98	50 ug/L	100 ug/L	2/98	1650 ug/kg	3300 ug/kg
Fluoranthene	206-44-0	SW846 8270C	9/98	0.90 ug/L	10 ug/L	2/98	51 ug/kg	330 ug/kg
Fluorene	86-73-7	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	57 ug/kg	330 ug/kg
Hexachlorobenzene	118-74-1	SW846 8270C	9/98	1.0 ug/L	10 ug/L	2/98	43 ug/kg	330 ug/kg
Hexachlorobutadiene	87-68-3	SW846 8270C	9/98	5.0 ug/L	10 ug/L	2/98	40 ug/kg	330 ug/kg
Hexachloro-1,3-cyclopentadiene	77-47-4	SW846 8270C	5/99	5 ug/L	50 ug/L	2/98	718 ug/kg	1600 ug/kg
Hexachloroethane	67-72-1	SW846 8270C	9/98	5.0 ug/L	10 ug/L	2/98	167 ug/kg	330 ug/kg
Hexachloropropene	1888-71-7	SW846 8270C	9/98	0.85 ug/L	10 ug/L	2/98	1650 ug/kg	3300 ug/kg
Indeno[1,2,3-cd]pyrene	193-39-5	SW846 8270C	9/98	0.85 ug/L	10 ug/L	2/98	18 ug/kg	330 ug/kg
Isodrin	465-73-6	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Isophorone	78-59-1	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	33 ug/kg	330 ug/kg
Isosafrole, Total	120-58-1	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	165 ug/kg	660 ug/kg
Kepone	143-50-0	SW846 8270C	9/98	50 ug/L	100 ug/L	2/98	1650 ug/kg	3300 ug/kg
Methapyrilene	91-80-5	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
2-Methylbenzenamine	95-53-4	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
3-Methylcholanthrene	56-49-5	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Methyl methanesulfonate	66-27-3	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
2-Methylnaphthalene	91-57-6	SW846 8270C	9/98	1.0 ug/L	10 ug/L	2/98	30 ug/kg	330 ug/kg
1-Methylnaphthalene	90-12-0	SW846 8270C	3/99	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Methyl parathion	298-00-0	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg

TABLE 5-11
SACRAMENTO LABORATORY
Method 8270C
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
2-Methylphenol	95-48-7	SW846 8270C	9/98	1.8 ug/L	10 ug/L	2/98	42 ug/kg	330 ug/kg
3-Methylphenol	108-39-4	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
4-Methylphenol	106-44-5	SW846 8270C	9/98	1.5 ug/L	10 ug/L	2/98	64 ug/kg	330 ug/kg
3-Methylphenol & 4-Methylphenol	65794-96-9	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Naphthalene	91-20-3	SW846 8270C	9/98	1.2 ug/L	10 ug/L	2/98	40 ug/kg	330 ug/kg
1,4-Naphthoquinone	130-15-4	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
1-Naphthylamine	134-32-7	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
2-Naphthylamine	91-59-8	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
2-Nitroaniline	88-74-4	SW846 8270C	9/98	0.68 ug/L	50 ug/L	2/98	285 ug/kg	1600 ug/kg
3-Nitroaniline	99-09-2	SW846 8270C	9/98	5.0 ug/L	50 ug/L	2/98	197 ug/kg	1600 ug/kg
4-Nitroaniline	100-01-6	SW846 8270C	9/98	0.81 ug/L	50 ug/L	2/98	504 ug/kg	1600 ug/kg
Nitrobenzene	98-95-3	SW846 8270C	9/98	1.1 ug/L	10 ug/L	2/98	35 ug/kg	330 ug/kg
2-Nitrophenol	88-75-5	SW846 8270C	9/98	2.0 ug/L	10 ug/L	2/98	55 ug/kg	330 ug/kg
4-Nitrophenol	100-02-7	SW846 8270C	9/98	10 ug/L	50 ug/L	2/98	720 ug/kg	1600 ug/kg
4-Nitroquinoline-1-oxide	56-57-5	SW846 8270C	9/98	50 ug/L	100 ug/L	4/98	1670 ug/kg	3300 ug/kg
N-Nitrosodi-n-butylamine	924-16-3	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitrosodiethylamine	55-18-5	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitrosodimethylamine	62-75-9	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitrosodiphenylamine	86-30-6	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitrosodi-n-propylamine	621-64-7	SW846 8270C	9/98	0.87 ug/L	10 ug/L	2/98	24 ug/kg	330 ug/kg
n-Nitrosomethylethylamine	10595-95-6	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitrosomorpholine	59-89-2	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitrosopiperidine	100-75-4	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitrosopyrrolidine	930-55-2	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
N-Nitro-o-toluidine	99-55-8	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Parathion	56-38-2	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
Pentachlorobenzene	608-93-5	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
Pentachloroethane	76-01-7	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
Pentachloronitrobenzene	82-68-8	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
Pentachlorophenol	87-86-5	SW846 8270C	9/98	18 ug/L	50 ug/L	2/98	250 ug/kg	1600 ug/kg
Phenacetin	62-44-2	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Phenanthrene	85-01-8	SW846 8270C	9/98	0.96 ug/L	10 ug/L	2/98	52 ug/kg	330 ug/kg
Phenol	108-95-2	SW846 8270C	9/98	5.0 ug/L	10 ug/L	2/98	59 ug/kg	330 ug/kg
p-Phenylene diamine	106-50-3	SW846 8270C	9/98	50 ug/L	100 ug/L	2/98	1650 ug/kg	3300 ug/kg
Phorate	298-02-2	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
2-Picoline	109-06-8	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Pronamide	23950-58-5	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Pyrene	129-00-0	SW846 8270C	9/98	1.0 ug/L	10 ug/L	2/98	58 ug/kg	330 ug/kg
Pyridine	110-86-1	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
Safrole, Total	94-59-7	SW846 8270C	9/98	10 ug/L	20 ug/L	2/98	330 ug/kg	660 ug/kg
1,2,4,5-Tetrachlorobenzene	95-94-3	SW846 8270C	9/98	5 ug/L	10 ug/L	2/98	165 ug/kg	330 ug/kg
2,3,4,6-Tetrachlorophenol	58-90-2	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
2,3,5,6-Tetrachlorophenol	935-95-5	SW846 8270C	3/99	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
Tetraethyldithiopyrophosphate	3689-24-5	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg

TABLE 5-11
SACRAMENTO LABORATORY
Method 8270C
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
(Sulfotepp)								
1,2,4-Trichlorobenzene	120-82-1	SW846 8270C	9/98	0.79 ug/L	10 ug/L	2/98	23 ug/kg	330 ug/kg
2,4,5-Trichlorophenol	95-95-4	SW846 8270C	9/98	1.7 ug/L	10 ug/L	2/98	112 ug/kg	330 ug/kg
2,4,6-Trichlorophenol	88-06-2	SW846 8270C	9/98	1.8 ug/L	10 ug/L	2/98	85 ug/kg	330 ug/kg
O,O,O-Triethylphosphorothioate	126-68-1	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg
1,3,5-Trinitrobenzene	99-35-4	SW846 8270C	9/98	25 ug/L	50 ug/L	2/98	800 ug/kg	1600 ug/kg

TABLE 5-12
SACRAMENTO LABORATORY
GC/MS Semi-Volatile CWM Chemical Degradate Method
Method Detection Limits (MDL) and
Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Acetophenone	98-86-2	SW846 8270C CWM	5/99	2.5 ug/L	5.0 ug/L	12/96	0.060 mg/kg	1.0 mg/kg
Benzothiazole	95-16-9	SW846 8270C CWM	12/96	0.476 ug/L	5.0 ug/L	12/96	0.097 mg/kg	1.0 mg/kg
Chloroacetophenone	532-27-4	SW846 8270C CWM	5/99	2.5 ug/L	5.0 ug/L	12/96	0.070 mg/kg	1.0 mg/kg
p-Chlorophenylmethylsulfide	13-09-1	SW846 8270C CWM	12/96	0.553 ug/L	5.0 ug/L	12/96	0.094 mg/kg	1.0 mg/kg
p-Chlorophenylmethylsulfone	98-57-7	SW846 8270C CWM	12/96	0.867 ug/L	10 ug/L	12/96	0.247 mg/kg	5.0 mg/kg
p-Chlorophenylmethylsulfoxide	934-73-6	SW846 8270C CWM	12/96	0.901 ug/L	10 ug/L	12/96	0.448 mg/kg	5.0 mg/kg
Chloropicrin	76-06-2	SW846 8270C CWM	5/99	2.5 ug/L	5.0 ug/L	12/96	0.30 mg/kg	1.0 mg/kg
Dimethyl disulfide	624-92-0	SW846 8270C CWM	12/96	0.266 ug/L	5.0 ug/L	12/96	0.046 mg/kg	0.5 mg/kg
1,4-Dithiane	505-29-3	SW846 8270C CWM	12/96	0.433 ug/L	5.0 ug/L	12/96	0.092 mg/kg	1.0 mg/kg
1,4-Oxathiane	15980-15-1	SW846 8270C CWM	12/96	0.572 ug/L	5.0 ug/L	12/96	0.074 mg/kg	0.5 mg/kg

TABLE 5-13
SACRAMENTO LABORATORY
Method 1668
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Decachlorobiphenyl	2051-24-3	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
Biphenyl, dichloro	25512-42-9	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
1,1'-Biphenyl, heptachloro-	28655-71-2	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
2,3,3',4,4',5,5'-Heptachlorobiphenyl	39635-31-9	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
1,1'-Biphenyl, 2,2',3,4,4',5,5'-heptachloro-	35065-29-3	EPA-14 1668	1/99	20 pg/L	20 pg/L	1/99	2.0 pg/g	2.0 pg/g
1,1'-Biphenyl, hexachloro-	26601-64-9	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
2,3',4,4',5,5'-Hexachlorobiphenyl	52663-72-6	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
2,3,3',4,4',5-Hexachlorobiphenyl	38380-08-4	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
2,3,3',4,4',5'-Hexachlorobiphenyl	69782-90-7	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
3,3',4,4',5,5'-Hexachlorobiphenyl	32774-16-6	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
Biphenyl, chloro	27323-18-8	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
1,1'-Biphenyl, nonachloro-	53742-07-7	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
1,1'-Biphenyl, octachloro-	55722-26-4	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
Biphenyl, pentachloro	25429-29-2	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
2',3,4,4',5-Pentachlorobiphenyl	65510-44-3	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
2,3',4,4',5-Pentachlorobiphenyl	31508-00-6	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
2,3,4,4',5-Pentachlorobiphenyl	74472-37-0	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
3,3',4,4',5-Pentachlorobiphenyl	57465-28-8	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
3,4,4',5-Tetrachlorobiphenyl	70362-50-4	EPA-14 1668	1/99	20 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
Tetrachlorobiphenyl	Q1180	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g
Biphenyl, 3,3',4,4'-tetrachloro	32598-13-3	EPA-14 1668	1/99	20.0 pg/L	20.0 pg/L	1/99	2.0 pg/g	2.0 pg/g
Biphenyl, trichloro	25323-68-6	EPA-14 1668	1/99	-- pg/L	pg/L	1/99	-- pg/g	pg/g

TABLE 5-14
SACRAMENTO LABORATORY
Method 1653
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Pentachlorophenol	87-86-5	EPA-20 1653	12/97	2.5 ug/L	5.0 ug/L		NR	NR
Tetrachlorocatechol	1198-55-6	EPA-20 1653	12/97	2.5 ug/L	5.0 ug/L		NR	NR
Tetrachloroguaiacol	2539-17-5	EPA-20 1653	12/97	2.5 ug/L	5.0 ug/L		NR	NR
2,3,4,6-Tetrachlorophenol	58-90-2	EPA-20 1653	12/97	1.25 ug/L	2.5 ug/L		NR	NR
3,4,5-Trichlorocatechol	56961-20-7	EPA-20 1653	12/97	2.5 ug/L	5.0 ug/L		NR	NR
3,4,6-Trichlorocatechol	32139-72-3	EPA-20 1653	12/97	2.5 ug/L	5.0 ug/L		NR	NR
4,5,6-Trichloroguaiacol	2668-24-8	EPA-20 1653	12/97	1.25 ug/L	2.5 ug/L		NR	NR
3,4,5-Trichloroguaiacol	57057-83-7	EPA-20 1653	12/97	1.25 ug/L	2.5 ug/L		NR	NR
3,4,6-Trichloroguaiacol	60712-44-9	EPA-20 1653	12/97	1.25 ug/L	2.5 ug/L		NR	NR
2,4,5-Trichlorophenol	95-95-4	EPA-20 1653	12/97	1.25 ug/L	2.5 ug/L		NR	NR
2,4,6-Trichlorophenol	88-06-2	EPA-20 1653	12/97	1.25 ug/L	2.5 ug/L		NR	NR
Trichlorosyringol	2539-26-6	EPA-20 1653	12/97	1.25 ug/L	2.5 ug/L		NR	NR

TABLE 5-15
SACRAMENTO LABORATORY
Method 1613B
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Total Hexachlorodibenzo-p-dioxins	34465-46-8	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
Total Hexachlorodibenzofurans	55684-94-1	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	35822-46-9	EPA-5 1613B	2/96	10.9 pg/L	pg/L	9/97	0.250 pg/g	pg/g
Total Heptachlorodibenzo-p-dioxins	37871-00-4	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4	EPA-5 1613B	2/96	18.9 pg/L	pg/L	9/97	0.250 pg/g	pg/g
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-89-7	EPA-5 1613B	2/96	16.3 pg/L	pg/L	9/97	0.250 pg/g	pg/g
Total Heptachlorodibenzofurans	38998-75-3	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	19408-74-3	EPA-5 1613B	2/96	15.9 pg/L	pg/L	9/97	0.338 pg/g	pg/g
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	39227-28-6	EPA-5 1613B	2/96	12.9 pg/L	pg/L	9/97	0.510 pg/g	pg/g
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	57653-85-7	EPA-5 1613B	2/96	17.0 pg/L	pg/L	9/97	0.348 pg/g	pg/g
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9	EPA-5 1613B	2/96	22.2 pg/L	pg/L	9/97	0.290 pg/g	pg/g
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9	EPA-5 1613B	2/96	12.0 pg/L	pg/L	9/97	2.50 pg/g	pg/g
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9	EPA-5 1613B	2/96	14.0 pg/L	pg/L	9/97	2.50 pg/g	pg/g
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5	EPA-5 1613B	2/96	15.8 pg/L	pg/L	9/97	2.50 pg/g	pg/g
Octachlorodibenzo-p-dioxin	3268-87-9	EPA-5 1613B	2/96	36.2 pg/L	pg/L	9/97	0.991 pg/g	pg/g
Octachlorodibenzofuran	39001-02-0	EPA-5 1613B	2/96	60.6 pg/L	pg/L	9/97	0.500 pg/g	pg/g
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	40321-76-4	EPA-5 1613B	2/96	12.5 pg/L	pg/L	9/97	0.250 pg/g	pg/g
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6	EPA-5 1613B	2/96	11.8 pg/L	pg/L	9/97	0.250 pg/g	pg/g
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4	EPA-5 1613B	2/96	13.8 pg/L	pg/L	9/97	0.250 pg/g	pg/g
Total Pentachlorodibenzo-p-dioxins	36088-22-9	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
Total Pentachlorodibenzofurans	30402-15-4	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	EPA-5 1613B	2/96	3.11 pg/L	pg/L	9/97	0.123 pg/g	pg/g
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	EPA-5 1613B	2/96	1.23 pg/L	pg/L	9/97	0.067 pg/g	pg/g
Total Tetrachlorodibenzo-p-dioxins	41903-57-5	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
Total Tetrachlorodibenzofurans	55722-27-5	EPA-5 1613B	3/99	0 pg/L	pg/L	3/99	0 pg/g	pg/g
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	EPA-5 1613B-Tetras	2/96	3.11 pg/L	pg/L	9/97	0.123 pg/g	NR
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	EPA-5 1613B-Tetras	2/96	1.23 pg/L	pg/L	9/97	0.067 pg/g	NR

TABLE 5-16
SACRAMENTO LABORATORY
Method 551
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	NCASI 551	2/96	3.11 pg/L	pg/L	7/97	0.123 pg/g	pg/g
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	NCASI 551	2/96	1.23 pg/L	pg/L	7/97	0.067 pg/g	pg/g

TABLE 5-17
SACRAMENTO LABORATORY
Chemical Degradate Methods
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Nitroguanidine	556-88-7	NONE UV/HPLC per SOP	10/97	0.960 ug/L	20 ug/L	10/97	0.023 mg/kg	0.25 mg/kg
Phosphorus	7723-14-0	SAC GC-FPD	4/98	0.0501 ug/L	1.0 ug/L	4/98	0.00058 mg/kg	0.010 mg/kg

TABLE 5-18
SACRAMENTO LABORATORY
Dioxin Method 8280
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Total Hexachlorodibenzo-p-dioxins	34465-46-8	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
Total Hexachlorodibenzofurans	55684-94-1	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	35822-46-9	SW846 8280	8/96	1.18 ng/L	ng/L	8/96	0.08 ng/g	ng/g
Total Heptachlorodibenzo-p-dioxins	37871-00-4	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4	SW846 8280	8/96	0.96 ng/L	ng/L	8/96	0.05 ng/g	ng/g
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-89-7	SW846 8280	8/96	0.83 ng/L	ng/L	8/96	0.09 ng/g	ng/g
Total Heptachlorodibenzofurans	38998-75-3	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	19408-74-3	SW846 8280	8/96	0.59 ng/L	ng/L	8/96	0.04 ng/g	ng/g
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	39227-28-6	SW846 8280	8/96	1.40 ng/L	ng/L	8/96	0.19 ng/g	ng/g
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	57653-85-7	SW846 8280	8/96	1.44 ng/L	ng/L	8/96	0.29 ng/g	ng/g
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9	SW846 8280	8/96	1.23 ng/L	ng/L	8/96	0.09 ng/g	ng/g
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9	SW846 8280	8/96	1.18 ng/L	ng/L	8/96	0.14 ng/g	ng/g
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9	SW846 8280	8/96	0.58 ng/L	ng/L	8/96	0.11 ng/g	ng/g
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5	SW846 8280	8/96	0.59 ng/L	ng/L	8/96	0.10 ng/g	ng/g
Octachlorodibenzo-p-dioxin	3268-87-9	SW846 8280	8/96	2.18 ng/L	ng/L	8/96	0.12 ng/g	ng/g
Octachlorodibenzofuran	39001-02-0	SW846 8280	8/96	1.10 ng/L	ng/L	8/96	0.12 ng/g	ng/g
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	40321-76-4	SW846 8280	8/96	0.86 ng/L	ng/L	8/96	0.08 ng/g	ng/g
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6	SW846 8280	8/96	1.17 ng/L	ng/L	8/96	0.12 ng/g	ng/g
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4	SW846 8280	8/96	0.87 ng/L	ng/L	8/96	0.08 ng/g	ng/g
Total Pentachlorodibenzo-p-dioxins	36088-22-9	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
Total Pentachlorodibenzofurans	30402-15-4	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	SW846 8280	8/96	0.30 ng/L	ng/L	8/96	0.30 ng/L	ng/g
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	SW846 8280	8/96	0.16 ng/L	ng/L	8/96	0.16 ng/L	ng/L
Total Tetrachlorodibenzo-p-dioxins	41903-57-5	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
Total Tetrachlorodibenzofurans	55722-27-5	SW846 8280	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g

TABLE 5-19
SACRAMENTO LABORATORY
Dioxin Method 8280A
Method Detection Limits (MDL) and
Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Total Hexachlorodibenzo-p-dioxins	34465-46-8	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
Total Hexachlorodibenzofurans	55684-94-1	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	35822-46-9	SW846 8280A	7/98	3.07 ng/L	ng/L	7/98	0.25 ng/g	ng/g
Total Heptachlorodibenzo-p-dioxins	37871-00-4	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4	SW846 8280A	7/98	1.25 ng/L	ng/L	7/98	0.16 ng/g	ng/g
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-89-7	SW846 8280A	7/98	5.51 ng/L	ng/L	5/98	0.35 ng/g	ng/g
Total Heptachlorodibenzofurans	38998-75-3	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	19408-74-3	SW846 8280A	7/98	5.20 ng/L	ng/L	5/98	0.48 ng/g	ng/g
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	39227-28-6	SW846 8280A	7/98	2.61 ng/L	ng/L	5/98	0.61 ng/g	ng/g
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	57653-85-7	SW846 8280A	7/98	3.53 ng/L	ng/L	5/98	0.83 ng/g	ng/g
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9	SW846 8280A	7/98	1.44 ng/L	ng/L	5/98	0.50 ng/g	ng/g
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9	SW846 8280A	7/98	3.73 ng/L	ng/L	5/98	0.63 ng/g	ng/g
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9	SW846 8280A	7/98	8.54 ng/L	ng/L	5/98	0.74 ng/g	ng/g
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5	SW846 8280A	7/98	3.62 ng/L	ng/L	5/98	0.28 ng/g	ng/g
Octachlorodibenzo-p-dioxin	3268-87-9	SW846 8280A	7/98	3.37 ng/L	ng/L	5/98	0.50 ng/g	ng/g
Octachlorodibenzofuran	39001-02-0	SW846 8280A	7/98	2.78 ng/L	ng/L	5/98	0.50 ng/g	ng/g
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	40321-76-4	SW846 8280A	7/98	2.64 ng/L	ng/L	5/98	0.29 ng/g	ng/g
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6	SW846 8280A	7/98	2.29 ng/L	ng/L	5/98	0.32 ng/g	ng/g
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4	SW846 8280A	7/98	1.44 ng/L	ng/L	5/98	0.39 ng/g	ng/g
Total Pentachlorodibenzo-p-dioxins	36088-22-9	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
Total Pentachlorodibenzofurans	30402-15-4	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	SW846 8280A	5/98	-- ng/L	ng/L	5/98	-- ng/L	ng/g
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	SW846 8280A	4/99	-- ng/L	ng/L	4/99	-- ng/L	ng/L
Total Tetrachlorodibenzo-p-dioxins	41903-57-5	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g
Total Tetrachlorodibenzofurans	55722-27-5	SW846 8280A	3/99	0 ng/L	ng/L	3/99	0 ng/g	ng/g

TABLE 5-20
SACRAMENTO LABORATORY
Dioxin Method 8290
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
	Q99999	SW846 8290		NR	NR	9/97	0.250 pg/g	NR
1,2,3,4,6,7,9-Heptachlorodibenzo-p-dioxin	58200-70-7	SW846 8290	3/99	0 pg/L	25 pg/L	3/99	2.5 pg/g	pg/g
1,2,3,4,6,7,9-Heptachlorodibenzofuran	70648-25-8	SW846 8290	3/99	0 pg/L	25 pg/L	3/99	2.5 pg/g	pg/g
Total Hexachlorodibenzo-p-dioxins	34465-46-8	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/L
Total Hexachlorodibenzofurans	55684-94-1	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/g
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	35822-46-9	SW846 8290	8/98	3.93 pg/L	pg/L	9/97	-- pg/L	pg/g
Total Heptachlorodibenzo-p-dioxins	37871-00-4	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4	SW846 8290	8/98	3.92 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-89-7	SW846 8290	8/98	5.08 pg/L	pg/L	9/97	-- pg/L	pg/L
Total Heptachlorodibenzofurans	38998-75-3	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	19408-74-3	SW846 8290	8/98	7.76 pg/L	pg/L	9/97	-- pg/L	pg/g
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	39227-28-6	SW846 8290	8/98	7.28 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	57653-85-7	SW846 8290	8/98	6.20 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9	SW846 8290	8/98	4.01 pg/L	pg/L	9/97	-- pg/L	pg/g
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9	SW846 8290	8/98	7.10 pg/L	pg/L	9/97	-- pg/L	pg/g
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9	SW846 8290	8/98	8.01 pg/L	pg/L	9/97	-- pg/L	pg/L
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5	SW846 8290	8/98	6.11 pg/L	pg/L	9/97	-- pg/L	pg/g
Octachlorodibenzo-p-dioxin	3268-87-9	SW846 8290	8/98	15.59 pg/L	pg/L	9/97	-- pg/L	pg/g
Octachlorodibenzofuran	39001-02-0	SW846 8290	8/98	11.88 pg/L	pg/L	9/97	-- pg/L	pg/g
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	40321-76-4	SW846 8290	8/98	6.59 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6	SW846 8290	8/98	6.00 pg/L	pg/L	9/97	-- pg/L	pg/L
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4	SW846 8290	8/98	4.90 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,3,4,7-Pentachlorodibenzo-p-dioxin	39227-61-7	SW846 8290	3/99	0 pg/L	pg/L	3/99	2.5 pg/g	pg/g
Total Pentachlorodibenzo-p-dioxins	36088-22-9	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/L
Total Pentachlorodibenzofurans	30402-15-4	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/L
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	SW846 8290	8/98	1.21 pg/L	pg/L	9/97	-- pg/L	pg/g
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	SW846 8290	8/98	1.92 pg/L	pg/L	9/97	-- pg/L	pg/L

TABLE 5-20
SACRAMENTO LABORATORY
Dioxin Method 8290
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
1,3,6,8-Tetrachlorodibenzo-p-dioxin	33423-92-6	SW846 8290	3/99	0 pg/L	5 pg/L	3/99	0.5 pg/g	pg/g
1,3,7,9-Tetrachlorodibenzo-p-dioxin	62470-53-5	SW846 8290	3/99	0 pg/L	5 pg/L	3/99	0.5 pg/g	pg/g
Total Tetrachlorodibenzo-p-dioxins	41903-57-5	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/L
1,2,7,8-Tetrachlorodibenzofuran	58802-20-3	SW846 8290	3/99	0 pg/L	5 pg/L	3/99	0.5 pg/g	pg/g
1,3,6,8-Tetrachlorodibenzofuran	71998-72-6	SW846 8290	3/99	0 pg/L	5 pg/L	3/99	0.5 pg/g	pg/g
Total Tetrachlorodibenzofurans	55722-27-5	SW846 8290	3/99	0 pg/L	pg/L	9/97	-- pg/L	pg/L

TABLE 5-21
SACRAMENTO LABORATORY
Method 8321
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Picric Acid	88-89-1	SW846 8321A	7/98	4.436 ug/L	20 ug/L	7/98	0.134 mg/kg	1.0 mg/kg
Thiodiglycol	111-48-8	SW846 8321-TDG	9/95	0.029 ug/L	0.25 ug/L	11/96	0.000618 mg/kg	0.025 mg/kg

TABLE 5-22
SACRAMENTO LABORATORY
Method 8321A
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
2-Amino-4,6-dinitrotoluene	35572-78-2	SW846 SW8321A	10/97	0.034 ug/L	0.26 ug/L	10/97	0.013 mg/kg	0.25 mg/kg
4-Amino-2,6-dinitrotoluene	19406-51-0	SW846 SW8321A	10/97	0.015 ug/L	0.26 ug/L	10/97	0.015 mg/kg	0.25 mg/kg
Bromacil	314-40-9	SW846 SW8321A	5/99	0.05 ug/L	0.1 ug/L	5/99	5.00 ug/kg	10 ug/kg
Diisopropylmethylphosphonate	1445-75-6	SW846 SW8321A	12/96	3.475 ug/L	10 ug/L	12/96	3.967 ug/kg	50 ug/kg
Dimethylmethylphosphonate	756-79-6	SW846 SW8321A	12/96	1.748 ug/L	10 ug/L	12/96	4.396 ug/kg	50 ug/kg
1,3-Dinitrobenzene	99-65-0	SW846 SW8321A	10/97	0.015 ug/L	4.0 ug/L	10/97	0.015 mg/kg	0.25 mg/kg
2,4-Dinitrotoluene	121-14-2	SW846 SW8321A	10/97	0.017 ug/L	0.26 ug/L	10/97	0.018 mg/kg	0.25 mg/kg
2,6-Dinitrotoluene	606-20-2	SW846 SW8321A	10/97	0.010 ug/L	0.26 ug/L	10/97	0.012 mg/kg	0.25 mg/kg
Diuron	330-54-1	SW846 SW8321A	5/99	0.05 ug/L	0.1 ug/L	5/99	5.00 ug/kg	10 ug/kg
Ethyl methylphosphonic acid	1832-53-7	SW846 SW8321A	12/96	2.936 ug/L	10 ug/L	12/96	4.872 ug/kg	50 ug/kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4	SW846 SW8321A	10/97	0.015 ug/L	14.0 ug/L	10/97	0.025 mg/kg	0.25 mg/kg
Isopropyl methyl phosphonic acid	1832-54-8	SW846 SW8321A	12/96	8.06 ug/L	50 ug/L	12/96	14.92 ug/kg	50 ug/kg
Methyl-2,4,6-trinitrophenylnitramine	479-45-8	SW846 SW8321A	10/97	0.009 ug/L	4.0 ug/L	10/97	0.009 mg/kg	0.25 mg/kg
[illegible]ethylphosphonic Acid	993-13-5	SW846 SW8321A	12/96	11.71 ug/L	100 ug/L	12/96	44.45 ug/kg	500 ug/kg
Nitrobenzene	98-95-3	SW846 SW8321A	10/97	0.100 ug/L	6.4 ug/L	10/97	0.100 mg/kg	0.25 mg/kg
Nitroglycerin	55-63-0	SW846 SW8321A	10/97	0.092 ug/L	10.0 ug/L	10/97	0.092 mg/kg	0.50 mg/kg
2&4-Nitrotoluene	Q1846	SW846 SW8321A	10/97	0.164 ug/L	0.26 ug/L	10/97	0.035 mg/kg	0.25 mg/kg
m-Nitrotoluene	99-08-1	SW846 SW8321A	10/97	0.136 ug/L	0.26 ug/L	10/97	0.136 mg/kg	0.25 mg/kg
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	SW846 SW8321A	10/97	0.115 ug/L	13.0 ug/L	10/97	0.115 mg/kg	0.50 mg/kg
Pentaerythritol Tetranitrate	78-11-5	SW846 SW8321A	10/97	0.069 ug/L	1.00 ug/L	4/98	0.115 mg/kg	0.50 mg/kg
Isalchem 123 Surfactant	Q1995	SW846 SW8321A	3/99	1.297 mg/L	5.0 mg/L	12/98	0.846 mg/kg	5.0 mg/kg
Thiodiglycol	111-48-8	SW846 SW8321A	9/95	0.029 ug/L	10 ug/L	12/96	4.208 ug/kg	50 ug/kg
1,3,5-Trinitrobenzene	99-35-4	SW846 SW8321A	10/97	0.010 ug/L	7.3 ug/L	10/97	0.010 mg/kg	0.25 mg/kg
2,4,6-Trinitrotoluene	118-96-7	SW846 SW8321A	10/97	0.005 ug/L	0.26 ug/L	10/97	0.005 mg/kg	0.25 mg/kg

TABLE 5-23
SACRAMENTO LABORATORY
Method 8331
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Tetrazene	31330-63-9	SW846 SW8331	11/98	0.809 ug/L	10 ug/L	11/98	0.099 ug/L	2.0 mg/kg

TABLE 5-24
SACRAMENTO LABORATORY
ICP MS Method 200.8
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Antimony	7440-36-0	MCAWW 200.8	4/98	0.00016 mg/L	0.002 mg/L	4/98	0.01 mg/kg	0.2 mg/kg
Arsenic	7440-38-2	MCAWW 200.8	4/98	0.00041 mg/L	0.002 mg/L	4/98	0.08 mg/kg	0.2 mg/kg
Barium	7440-39-3	MCAWW 200.8	4/98	0.00043 mg/L	0.001 mg/L	4/98	0.04 mg/kg	0.1 mg/kg
Beryllium	7440-41-7	MCAWW 200.8	4/98	0.00011 mg/L	0.001 mg/L	4/98	0.01 mg/kg	0.1 mg/kg
Bismuth	7440-69-9	MCAWW 200.8	4/99	0.005 mg/L	0.005 mg/L		NR	NR
Cadmium	7440-43-9	MCAWW 200.8	4/98	0.0001 mg/L	0.001 mg/L	4/98	0.01 mg/kg	0.1 mg/kg
Chromium, Total	7440-47-3	MCAWW 200.8	4/98	0.00035 mg/L	0.002 mg/L	4/98	0.047 mg/kg	0.2 mg/kg
Cobalt	7440-48-4	MCAWW 200.8	4/98	0.0001 mg/L	0.001 mg/L	4/98	0.01 mg/kg	0.1 mg/kg
Copper	7440-50-8	MCAWW 200.8	4/98	0.0015 mg/L	0.002 mg/L	4/98	0.011 mg/kg	0.2 mg/kg
Lead	7439-92-1	MCAWW 200.8	4/98	0.001 mg/L	0.001 mg/L	4/98	0.01 mg/kg	0.1 mg/kg
Mercury	7439-97-6	MCAWW 200.8	4/98	0.0002 mg/L	0.0002 mg/L	4/98	0.01 mg/kg	0.04 mg/kg
Molybdenum	7439-98-7	MCAWW 200.8	4/98	0.0001 mg/L	0.001 mg/L	4/98	0.01 mg/kg	0.1 mg/kg
Nickel	7440-02-0	MCAWW 200.8	4/98	0.00012 mg/L	0.002 mg/L	4/98	0.01 mg/kg	0.2 mg/kg
Selenium	7782-49-2	MCAWW 200.8	4/98	0.00012 mg/L	0.002 mg/L	4/98	0.025 mg/kg	0.2 mg/kg
Silver	7440-22-4	MCAWW 200.8	4/98	0.0001 mg/L	0.001 mg/L	4/98	0.01 mg/kg	0.1 mg/kg
Thallium	7440-28-0	MCAWW 200.8	4/98	0.0001 mg/L	0.001 mg/L	4/98	0.036 mg/kg	0.1 mg/kg
Vanadium	7440-62-2	MCAWW 200.8	4/98	0.00034 mg/L	0.01 mg/L	4/98	0.43 mg/kg	1.0 mg/kg
Zinc	7440-66-6	MCAWW 200.8	4/98	0.0011 mg/L	0.002 mg/L	4/98	0.03 mg/kg	0.2 mg/kg

TABLE 5-25
SACRAMENTO LABORATORY
Mercury Methods
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Mercury	7439-97-6	SW846 7470A	6/98	0.000049 mg/L	0.0002 mg/L		NR	NR
Mercury	7439-97-6	SW846 7471A		NR	NR	7/98	0.005 mg/kg	0.04 mg/kg
Mercury	7439-97-6	MCAWW 245.1	6/98	0.000049 mg/L	0.0002 mg/L		NR	NR

TABLE 5-26
SACRAMENTO LABORATORY
ICP Trace Method 6010B
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Antimony	7440-36-0	SW846 6010B	3/98	0.0022 mg/L	0.01 mg/L	3/98	0.16 mg/kg	1.0 mg/kg
Arsenic	7440-38-2	SW846 6010B	3/98	0.0042 mg/L	0.01 mg/L	3/98	0.28 mg/kg	1.0 mg/kg
Barium	7440-39-3	SW846 6010B	3/98	0.00014 mg/L	0.01 mg/L	3/98	0.02 mg/kg	1.0 mg/kg
Beryllium	7440-41-7	SW846 6010B	3/98	0.00005 mg/L	0.005 mg/L	3/98	0.01 mg/kg	0.5 mg/kg
Cadmium	7440-43-9	SW846 6010B	3/98	0.00037 mg/L	0.005 mg/L	3/98	0.03 mg/kg	0.5 mg/kg
Chromium, Total	7440-47-3	SW846 6010B	3/98	0.00051 mg/L	0.005 mg/L	3/98	0.08 mg/kg	0.5 mg/kg
Cobalt	7440-48-4	SW846 6010B	3/98	0.00088 mg/L	0.05 mg/L	3/98	0.09 mg/kg	5.0 mg/kg
Copper	7440-50-8	SW846 6010B	3/98	0.00062 mg/L	0.025 mg/L	3/98	0.09 mg/kg	2.5 mg/kg
Iron	7439-89-6	SW846 6010B	4/98	0.10 mg/L	0.10 mg/L	3/98	1.26 mg/kg	10 mg/kg
Lead	7439-92-1	SW846 6010B	3/98	0.0018 mg/L	0.005 mg/L	3/98	0.15 mg/kg	0.5 mg/kg
Manganese	7439-96-5	SW846 6010B	3/98	0.00047 mg/L	0.015 mg/L	3/98	0.01 mg/kg	1.5 mg/kg
Molybdenum	7439-98-7	SW846 6010B	3/98	0.0015 mg/L	0.04 mg/L	3/98	0.16 mg/kg	4.0 mg/kg
Nickel	7440-02-0	SW846 6010B	3/98	0.0012 mg/L	0.04 mg/L	3/98	0.07 mg/kg	4.0 mg/kg
Selenium	7782-49-2	SW846 6010B	3/98	0.0032 mg/L	0.005 mg/L	3/98	0.29 mg/kg	0.5 mg/kg
Silver	7440-22-4	SW846 6010B	3/98	0.00099 mg/L	0.005 mg/L	3/98	0.11 mg/kg	0.5 mg/kg
Thallium	7440-28-0	SW846 6010B	3/98	0.003 mg/L	0.01 mg/L	3/98	0.39 mg/kg	1.0 mg/kg
Vanadium	7440-62-2	SW846 6010B	3/98	0.00089 mg/L	0.05 mg/L	3/98	0.04 mg/kg	5.0 mg/kg
Zinc	7440-66-6	SW846 6010B	3/98	0.0024 mg/L	0.02 mg/L	3/98	0.06 mg/kg	2.0 mg/kg

TABLE 5-27
SACRAMENTO LABORATORY
ICP Method 6010B
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Aluminum	7429-90-5	SW846 6010B	4/98	0.043 mg/L	0.20 mg/L	3/98	2.4 mg/kg	20 mg/kg
Antimony	7440-36-0	SW846 6010B	4/98	0.031 mg/L	0.06 mg/L	3/98	2.2 mg/kg	6.0 mg/kg
Arsenic	7440-38-2	SW846 6010B	4/98	0.043 mg/L	0.30 mg/L	3/98	3.1 mg/kg	30 mg/kg
Barium	7440-39-3	SW846 6010B	4/98	0.00043 mg/L	0.20 mg/L	3/98	0.04 mg/kg	20 mg/kg
Beryllium	7440-41-7	SW846 6010B	4/98	0.00009 mg/L	0.005 mg/L	3/98	0.01 mg/kg	0.5 mg/kg
Boron	7440-42-8	SW846 6010B	4/98	0.011 mg/L	0.20 mg/L	3/98	0.40 mg/kg	20 mg/kg
Cadmium	7440-43-9	SW846 6010B	4/98	0.0031 mg/L	0.005 mg/L	3/98	0.44 mg/kg	0.5 mg/kg
Calcium	7440-70-2	SW846 6010B	4/98	0.027 mg/L	5.0 mg/L	3/98	1.2 mg/kg	500 mg/kg
Chromium, Total	7440-47-3	SW846 6010B	4/98	0.0028 mg/L	0.01 mg/L	3/98	0.27 mg/kg	1.0 mg/kg
Cobalt	7440-48-4	SW846 6010B	4/98	0.0074 mg/L	0.05 mg/L	3/98	0.89 mg/kg	5.0 mg/kg
Copper	7440-50-8	SW846 6010B	4/98	0.0021 mg/L	0.025 mg/L	3/98	0.39 mg/kg	2.5 mg/kg
Iron	7439-89-6	SW846 6010B	4/98	0.0039 mg/L	0.1 mg/L	3/98	0.09 mg/kg	10 mg/kg
Lead	7439-92-1	SW846 6010B	4/98	0.031 mg/L	0.10 mg/L	3/98	2.2 mg/kg	10 mg/kg
Lithium	7439-93-2	SW846 6010B	4/98	0.0026 mg/L	0.05 mg/L	3/98	0.23 mg/kg	5.0 mg/kg
Magnesium	7439-95-4	SW846 6010B	4/98	0.025 mg/L	5.0 mg/L	3/98	1.8 mg/kg	500 mg/kg
Manganese	7439-96-5	SW846 6010B	4/98	0.00069 mg/L	0.015 mg/L	3/98	0.30 mg/kg	1.5 mg/kg
Molybdenum	7439-98-7	SW846 6010B	4/98	0.0046 mg/L	0.04 mg/L	3/98	0.51 mg/kg	4.0 mg/kg
Nickel	7440-02-0	SW846 6010B	4/98	0.016 mg/L	0.04 mg/L	3/98	1.8 mg/kg	4.0 mg/kg
Phosphorus	7723-14-0	SW846 6010B	4/98	0.12 mg/L	0.3 mg/L	3/98	12 mg/kg	30 mg/kg
Potassium	7440-09-7	SW846 6010B	4/98	0.6 mg/L	5.0 mg/L	3/98	103 mg/kg	500 mg/kg
Selenium	7782-49-2	SW846 6010B	4/98	0.051 mg/L	0.25 mg/L	3/98	7.5 mg/kg	25 mg/kg
Silicon	7440-21-3	SW846 6010B	4/98	0.005 mg/L	0.5 mg/L	3/98	1.8 mg/kg	50 mg/kg
Silver	7440-22-4	SW846 6010B	4/98	0.0032 mg/L	0.01 mg/L	3/98	0.20 mg/kg	1.0 mg/kg
Sodium	7440-23-5	SW846 6010B	4/98	0.036 mg/L	5.0 mg/L	3/98	2.8 mg/kg	500 mg/kg
Strontium	7440-24-6	SW846 6010B	4/98	0.00033 mg/L	0.05 mg/L	3/98	0.02 mg/kg	5 mg/kg
Sulfur	7704-34-9	SW846 6010B	4/98	0.035 mg/L	0.1 mg/L	3/98	3.8 mg/kg	10 mg/kg
Thallium	7440-28-0	SW846 6010B	4/98	0.053 mg/L	2.0 mg/L	3/98	7.1 mg/kg	200 mg/kg
Tin	7440-31-5	SW846 6010B	4/98	0.022 mg/L	0.10 mg/L	3/98	2.9 mg/kg	10 mg/kg
Titanium	7440-32-6	SW846 6010B	4/98	0.0017 mg/L	0.05 mg/L	3/98	0.25 mg/kg	5.0 mg/kg
Vanadium	7440-62-2	SW846 6010B	4/98	0.0032 mg/L	0.05 mg/L	3/98	0.25 mg/kg	5.0 mg/kg
Zinc	7440-66-6	SW846 6010B	4/98	0.0021 mg/L	0.02 mg/L	3/98	0.40 mg/kg	2.0 mg/kg

TABLE 5-28
SACRAMENTO LABORATORY
ICP MS Method 6020
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Aluminum	7429-90-5	SW846 6020	6/98	0.022 mg/L	0.05 mg/L	6/98	0.16 mg/kg	5.0 mg/kg
Antimony	7440-36-0	SW846 6020	6/98	0.00016 mg/L	0.002 mg/L	6/98	0.01 mg/kg	0.2 mg/kg
Arsenic	7440-38-2	SW846 6020	6/98	0.00041 mg/L	0.002 mg/L	6/98	0.08 mg/kg	0.2 mg/kg
Barium	7440-39-3	SW846 6020	6/98	0.00043 mg/L	0.001 mg/L	6/98	0.04 mg/kg	0.1 mg/kg
Beryllium	7440-41-7	SW846 6020	6/98	0.00011 mg/L	0.001 mg/L	6/98	0.01 mg/kg	0.1 mg/kg
Bismuth	7440-69-9	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Boron	7440-42-8	SW846 6020	6/98	0.0093 mg/L	0.05 mg/L	6/98	0.5 mg/kg	5.0 mg/kg
Cadmium	7440-43-9	SW846 6020	6/98	0.0001 mg/L	0.001 mg/L	6/98	0.01 mg/kg	0.1 mg/kg
Calcium	7440-70-2	SW846 6020	3/98	0.0032 mg/L	0.05 mg/L	6/98	0.84 mg/kg	5.0 mg/kg
Cerium	7440-45-1	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Cesium	7440-46-2	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Chromium, Total	7440-47-3	SW846 6020	6/98	0.00035 mg/L	0.002 mg/L	6/98	0.047 mg/kg	0.2 mg/kg
Cobalt	7440-48-4	SW846 6020	6/98	0.0001 mg/L	0.001 mg/L	6/98	0.01 mg/kg	0.1 mg/kg
Copper	7440-50-8	SW846 6020	3/98	0.00015 mg/L	0.002 mg/L	6/98	0.011 mg/kg	0.2 mg/kg
Dysprosium	7429-91-6	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Erbium	7440-52-0	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Europium	7440-53-1	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Gadolinium	7440-54-2	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Gallium	7440-55-3	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Germanium	7440-56-4	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/98	0.025 mg/kg	0.5 mg/kg
Gold	7440-57-5	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Hafnium	7440-58-6	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Holmium	7440-60-0	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Iridium	7439-88-5	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Iron	7439-89-6	SW846 6020	6/98	0.0035 mg/L	0.05 mg/L	6/98	1.1 mg/kg	5.0 mg/kg
Lanthanum	7439-91-0	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Lead	7439-92-1	SW846 6020	6/98	0.00015 mg/L	0.001 mg/L	6/98	0.01 mg/kg	0.1 mg/kg
Lithium	7439-93-2	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Lutetium	7439-94-3	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Magnesium	7439-95-4	SW846 6020	6/98	0.0016 mg/L	0.05 mg/L	6/98	0.11 mg/kg	5.0 mg/kg
Manganese	7439-96-5	SW846 6020	6/98	0.00014 mg/L	0.001 mg/L	6/98	0.01 mg/kg	0.1 mg/kg
Mercury	7439-97-6	SW846 6020	4/98	0.0002 mg/L	0.0002 mg/L	6/98	0.01 mg/kg	0.02 mg/kg
Molybdenum	7439-98-7	SW846 6020	6/98	0.0001 mg/L	0.001 mg/L	6/98	0.01 mg/kg	0.1 mg/kg
Neodymium	7440-00-8	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Nickel	7440-02-0	SW846 6020	6/98	0.00012 mg/L	0.002 mg/L	6/98	0.01 mg/kg	0.2 mg/kg
Niobium	7440-03-1	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Palladium	7440-05-3	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Phosphorus	7723-14-0	SW846 6020	4/98	0.032 mg/L	0.05 mg/L	6/98	1.7 mg/kg	5.0 mg/kg
Platinum	7440-06-4	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Potassium	7440-09-7	SW846 6020	6/98	0.0024 mg/L	0.05 mg/L	6/98	0.5 mg/kg	5.0 mg/kg
Praseodymium	7440-10-0	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Rhenium	7440-15-5	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Rhodium	7440-16-6	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Rubidium	7440-17-7	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg

TABLE 5-28
SACRAMENTO LABORATORY
ICP MS Method 6020
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Ruthenium	7440-18-8	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Samarium	7440-19-9	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Scandium	7440-20-2	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Selenium	7782-49-2	SW846 6020	3/98	0.00012 mg/L	0.002 mg/L	6/98	0.025 mg/kg	0.2 mg/kg
Silver	7440-22-4	SW846 6020	6/98	0.0001 mg/L	0.001 mg/L	6/98	0.01 mg/kg	0.1 mg/kg
Sodium	7440-23-5	SW846 6020	6/98	0.016 mg/L	0.05 mg/L	6/98	0.68 mg/kg	5.0 mg/kg
Strontium	7440-24-6	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Tantalum	7440-25-7	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Tellurium	13494-80-9	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Terbium	7440-27-9	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Thallium	7440-28-0	SW846 6020	3/98	0.0001 mg/L	0.001 mg/L	6/98	0.036 mg/kg	0.1 mg/kg
Thorium	7440-29-1	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Thulium	7440-30-4	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Tin	7440-31-5	SW846 6020	6/98	0.00034 mg/L	0.01 mg/L	6/98	0.1 mg/kg	1.0 mg/kg
Titanium	7440-32-6	SW846 6020	3/98	0.00039 mg/L	0.002 mg/L	6/98	0.019 mg/kg	0.2 mg/kg
Tungsten	7440-33-7	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Uranium	7440-61-1	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Vanadium	7440-62-2	SW846 6020	6/98	0.00034 mg/L	0.01 mg/L	6/98	0.43 mg/kg	1.0 mg/kg
Ytterbium	7440-64-4	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Yttrium	Q713	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Zinc	7440-66-6	SW846 6020	5/99	0.0011 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg
Zirconium	7440-67-7	SW846 6020	6/99	0.001 mg/L	0.005 mg/L	6/99	0.1 mg/kg	0.5 mg/kg

TABLE 5-29
SACRAMENTO LABORATORY
Graphite Furnace Methods 7000
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
Arsenic	7440-38-2	SW846 7060A	9/98	0.002 mg/L	0.01 mg/L	3/98	0.24 mg/kg	1.0 mg/kg
Lead	7439-92-1	SW846 7421	9/98	0.0014 mg/L	0.005 mg/L	3/98	0.093 mg/kg	0.5 mg/kg
Nickel	7440-02-0	SW846 7521	5/99	0.0015 mg/L	0.005 mg/L		NR	NR
Selenium	7782-49-2	SW846 7740	9/98	0.0017 mg/L	0.005 mg/L	3/98	0.17 mg/kg	0.5 mg/kg
Thallium	7440-28-0	SW846 7841	9/98	0.0032 mg/L	0.01 mg/L	3/98	0.21 mg/kg	1.0 mg/kg

TABLE 5-30
SACRAMENTO LABORATORY
Oil and Grease Methods
Method Detection Limits (MDL) and Reporting Limits (RL)

Parameter	CAS No.	Method	Water			Soil		
			Date	MDL	RL	Date	MDL	RL
n-Hexane Extractable Material	Q1250	CFR136A 1664 HEM	10/98	1.35 mg/L	5.0 mg/L	12/98	54.6 mg/kg	166.7 mg/kg
n-Hexane Extractable Material, Silica Gel Treated	Q1250	CFR136A 1664 SGT HEM	11/98	0.59 mg/L	10.0 mg/L	12/98	18.65 mg/kg	333.3 mg/kg

TABLE 5-31
SACRAMENTO LABORATORY
General Chemistry Methods
Method Detection Limits (MDL) and Reporting Limits (RL)

			Water			Soil		
Parameter	CAS No.	Method	Date	MDL	RL	Date	MDL	RL
HALOGENS,TOTAL ADSORBABLE	Q1874	EPA-17 1650A AOX	12/98	4.75 ug/L	20 ug/L		NR	NR
Cyanide, Total	57-12-5	ICLP ILM04.0	1/99	0.002 mg/L	0.01 mg/L	1/99	0.092 mg/kg	0.50 mg/kg
Conductivity	Q181	MCAWW 120.1	1/99	2.0 umhos/cm	2.0 umhos/cm	2/98	---- umhos/cm	2.0 umhos/cm
Hardness, as CaCO3	Q356	MCAWW 130.2	12/98	2.5 mg/L	5.0 mg/L		NR	NR
Solids (Residue) Filterable	Q594	MCAWW 160.1	1/99	10 mg/L	10 mg/L		NR	NR
Solids (Residue)Non-Filterable	Q595	MCAWW 160.2	7/98	5.0 mg/L	5 mg/L		NR	NR
Solids (Residue) Total	Q597	MCAWW 160.3	1/99	10 mg/L	10 mg/L		NR	NR
Solids (Residue) Settleable, Volumetric	Q596	MCAWW 160.5	1/99	0.50 mL/L/hr	0.50 mL/L/hr		NR	NR
Turbidity	Q671	MCAWW 180.1	2/98	0.17 NTU	1.0 NTU		NR	NR
Bromide	Q85	MCAWW 300.0A	12/98	0.05 mg/L	0.5 mg/L	12/98	0.25 mg/kg	2.5 mg/kg
Chloride	Q138	MCAWW 300.0A	12/98	0.10 mg/L	1.0 mg/L	12/98	0.50 mg/kg	5.0 mg/kg
Fluoride	Q338	MCAWW 300.0A	12/98	0.051 mg/L	0.50 mg/L	12/98	0.255 mg/kg	2.5 mg/kg
Nitrogen, Nitrate	Q479	MCAWW 300.0A	12/98	0.005 mg/L	0.05 mg/L	12/98	0.025 mg/kg	0.25 mg/kg
Nitrogen, Nitrite	Q481	MCAWW 300.0A	12/98	0.007 mg/L	0.05 mg/L	12/98	0.035 mg/kg	0.25 mg/kg
Phosphate as P, Ortho	Q551	MCAWW 300.0A	12/98	0.055 mg/L	0.2 mg/L	12/98	0.275 mg/kg	1.0 mg/kg
Sulfate	Q605	MCAWW 300.0A	12/98	0.10 mg/L	1.0 mg/L	12/98	0.50 mg/kg	5.0 mg/kg
Alkalinity, Total, as CaCO3	Q18	MCAWW 310.1	1/99	5.0 mg/L	5 mg/L		NR	25 mg/kg
Chloride	Q138	MCAWW 325.2	12/98	0.355 mg/L	1.0 mg/L		NR	NR
Chloride	Q138	MCAWW 325.3	12/98	1.7 mg/L	5.0 mg/L		NR	NR
Cyanide, Total	57-12-5	MCAWW 335.4	1/99	0.002 mg/L	0.01 mg/L	1/99	0.092 mg/kg	0.50 mg/kg
Fluoride	Q338	MCAWW 340.2	12/98	0.02 mg/L	0.10 mg/L		NR	NR
Ammonia	7664-41-7	MCAWW 350.1	12/98	0.036 mg/L	0.10 mg/L	12/98	0.18 mg/kg	0.50 mg/kg
Nitrogen, Total Kjeldahl	Q483	MCAWW 351.2	12/98	0.20 mg/L	0.50 mg/L	12/98	4.18 mg/kg	10 mg/kg
Nitrogen, Total Organic	Q484	MCAWW 351.2/350.1	1/99	-- mg/L	0.50 mg/L		NR	NR
Nitrocellulose	9004-70-0	MCAWW 353.2	11/97	0.367 mg/L	0.50 mg/L	11/97	0.276 mg/kg	2.0 mg/kg
Nitrogen, Nitrate	Q479	MCAWW 353.2	12/98	0.023 mg/L	0.05 mg/L		NR	NR
Nitrogen, Nitrate/Nitrite	Q480	MCAWW 353.2	12/98	0.015 mg/L	0.05 mg/L		NR	NR
Nitrogen, Nitrite	Q481	MCAWW 353.2	12/98	0.020 mg/L	0.05 mg/L		NR	NR
Phosphate as P, Ortho	Q551	MCAWW 365.3	12/98	0.004 mg/L	0.05 mg/L	12/98	0.020 mg/kg	0.25 mg/kg
Phosphorus	7723-14-0	MCAWW 365.3		NR	NR	11/97	1.6 mg/kg	NR
Phosphorus	7723-14-0	MCAWW 365.3	12/98	0.007 mg/L	0.050 mg/L	12/98	0.70 mg/kg	5.0 mg/kg
Phosphorus, Organic	7723-14-0	MCAWW 365.3	4/99	0.025 mg/L	0.05 mg/L		NR	NR
Sulfate	Q605	MCAWW 575.4	11/98	2.15 mg/L	5.0 mg/L	4/99	12.5 mg/kg	25 mg/kg
Sulfide, Total	Q608	MCAWW 376.2	10/97	0.014 mg/L	0.050 mg/L	10/97	0.070 mg/kg	0.25 mg/kg
Chemical Oxygen Demand	Q132	MCAWW 410.4	12/98	7.2 mg/L	10 mg/L		NR	NR
Carbon, Dissolved Organic	Q23	MCAWW 415.1	12/98	0.11 mg/L	1.0 mg/L		NR	NR
Carbon, Total Inorganic	Q129	MCAWW 415.1	2/98	0.025 mg/L	1.0 mg/L		NR	NR
Total Organic Carbon	7440-44-0	MCAWW 415.1	12/98	0.11 mg/L	1.0 mg/L	5/97	24.5 mg/kg	NR
Phenolics, Total Recoverable	Q540	MCAWW 420.4	1/99	0.003 mg/L	0.01 mg/L	12/98	0.161 mg/kg	0.50 mg/kg
Alkalinity, bicarbonate, as CaCO3	Q17	SM18 2320 B	1/99	5.0 mg/L	5.0 mg/L		NR	NR
Alkalinity, carbonate, as CaCO3	Q22	SM18 2320 B	1/99	5.0 mg/L	5.0 mg/L		NR	NR

TABLE 5-31
SACRAMENTO LABORATORY
General Chemistry Methods
Method Detection Limits (MDL) and Reporting Limits (RL)

Parameter	CAS No.	Method	Water			Soil		
			Date	MDL	RL	Date	MDL	RL
Alkalinity, hydroxide, as CaCO3	Q21	SM18 2320 B	1/99	5.0 mg/L	5.0 mg/L		NR	NR
Alkalinity, phenolphthalein, as CaCO3	Q19	SM18 2320 B	1/99	5.0 mg/L	5.0 mg/L		NR	NR
Alkalinity, Total, as CaCO3	Q18	SM18 2320 B	1/99	5.0 mg/L	5.0 mg/L		NR	NR
Chromium, hexavalent	18540-29-9	SM18 3500-CR D	12/98	0.002 mg/L	0.010 mg/L		NR	NR
Cyanide, Total	57-12-5	SM18 4500-CN-I	1/99	0.01 mg/L	0.01 mg/L	1/99	0.50 mg/kg	0.50 mg/kg
Total Organic Carbon	7440-44-0	SM18 5310C	12/98	0.11 mg/L	1.0 mg/L		NR	NR
Chromium, hexavalent	18540-29-9	SW846 7196A	12/98	0.002 mg/L	0.01 mg/L	12/98	0.010 mg/kg	0.05 mg/kg
Cyanide, Amenable	Q188	SW846 9012A	1/99	0.01 mg/L	0.01 mg/L	1/99	0.50 mg/kg	0.50 mg/kg
Cyanide, Total	57-12-5	SW846 9012A	1/99	0.002 mg/L	0.01 mg/L	1/99	0.092 mg/kg	0.50 mg/kg
Halogens, Total Organic	Q355	SW846 9020B	12/98	3.97 ug/L	30 ug/L		NR	NR
Conductivity	Q181	SW846 9050A	1/99	2.0 umhos/cm	2.0 umhos/cm		NR	NR
Bromide	Q85	SW846 9056	12/98	0.05 mg/L	0.5 mg/L		NR	NR
Chloride	Q138	SW846 9056	12/98	0.10 mg/L	1.0 mg/L		NR	NR
Fluoride	Q338	SW846 9056	12/98	0.051 mg/L	0.50 mg/L		NR	NR
Nitrogen, Nitrate	Q479	SW846 9056	12/98	0.005 mg/L	0.05 mg/L		NR	NR
Nitrogen, Nitrite	Q481	SW846 9056	12/98	0.007 mg/L	0.05 mg/L		NR	NR
Phosphate as P, Ortho	Q551	SW846 9056	12/98	0.055 mg/L	0.2 mg/L		NR	NR
Sulfate	Q605	SW846 9056	12/98	0.10 mg/L	1.0 mg/L		NR	NR
Carbon, Dissolved Organic	Q23	SW846 9060	12/98	0.11 mg/L	1.0 mg/L		NR	NR
Carbon, Total	Q122	SW846 9060		NR	NR	2/99	50 mg/kg	100 mg/kg
Carbon, Total Inorganic	Q129	SW846 9060		NR	NR	2/99	100 mg/kg	100 mg/kg
Total Organic Carbon	7440-44-0	SW846 9060	12/98	0.11 mg/L	1.0 mg/L	1/99	76.8 mg/kg	100 mg/kg
Phenolics, Total Recoverable	Q540	SW846 9066	1/99	0.003 mg/L	0.01 mg/L	12/98	0.161 mg/kg	0.50 mg/kg
Chloride	Q138	SW846 9251	12/98	0.355 mg/L	1.0 mg/L		NR	NR

Facility Appendix
Sacramento Laboratory

Section Sacramento-6

Performance
Evaluation
Studies

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TABLE 6-1
SACRAMENTO LABORATORY
Performance Evaluation Sample Programs

Performance Evaluation Sample Program Description	Analysis Performed	Frequency of Participation
Water Pollution Laboratory PE Study, Environmental Resource Associates	Trace Metals, Minerals, Nutrients, Demand, PCBs in Water, PCBs in Transformer Oil, Pesticides (Insecticides), Volatile Halocarbons, Volatile Aromatics, and Miscellaneous (TSS, Cyanide, Oil & Grease, Total Phenolics), Base Neutrals/Acids, Total Petroleum Hydrocarbons	Semi-annually
Water Supply Laboratory PE Study, Environmental Resource Associates	Trace Metals, Nitrate/Nitrite/Fluoride, Pesticides, Herbicides, PAHs, Trihalomethanes, Regulated VOCs, and Miscellaneous (TDS, Hardness, pH, Alkalinity, Sodium, Sulfate, Cyanide).	Semi-annually
USACE-MRD	Metals, Anions, Phenolics, Cyanide, Volatiles by GC and GCMS, Organochlorine, Pesticides, PCBs, Herbicides, PAH, TOC, TPH Gas and Diesel, Explosives, Dioxins, Semivoltiles by GCMS	18 Months
Accustandard	2,3,7,8-TCDD	Semi-annually
USGS	Metals, General Chemistry	Annually
Quanterra Inc. Double-Blind	Volatile Organics, Metals, General Chemistry, Base/Neutral Extractables, Acid Extractables, Project Managemet	Annually
New York State Dept. of Conservation	PCB Congeners by 1668	Annually
Analytical Products Group, Inc. (APG) Real World Matrix PE Program	Demand, Nutrients, Solids, Oil and Grease, Minerals, pH, Trace Metals, Phenol, Cyanide, Fluoride, Total Organic Halide, Hexavalent Chromium, Volatile Organics, Acid Extractables, Base/Neutral Extractables, Pesticides, Polynuclear Aromatic Hydrocarbons, Purgeable Aromatics (BTEX)	Semi-annually

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Facility Appendix
Sacramento Laboratory

Section Sacramento-7

Additional
Operation-Specific
Information

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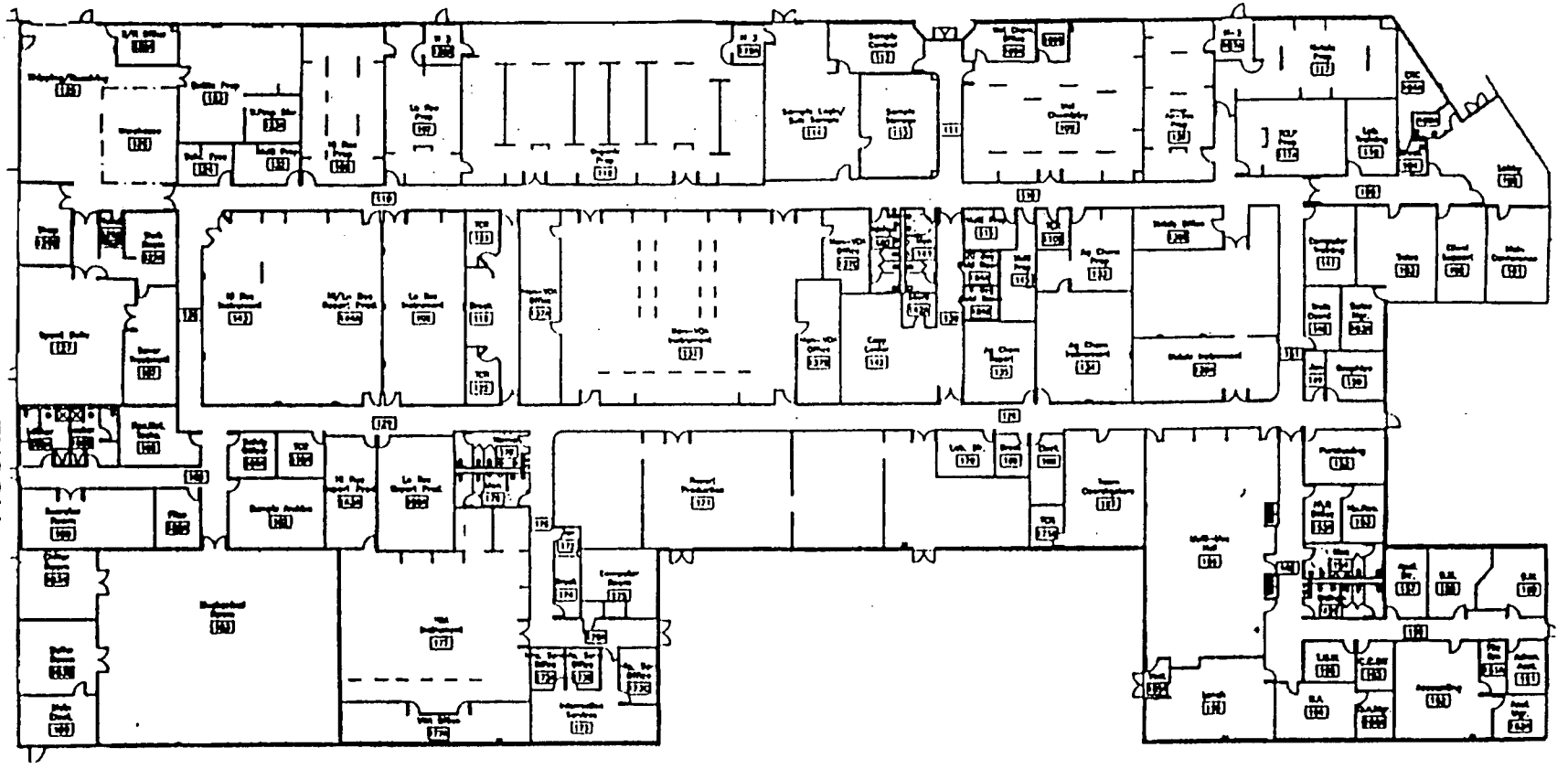
FIGURE 7-1



KORNBERG ASSOCIATES
Architects
DEL MAR, CA (619) 755-6390
MENLO PARK, CA (415) 321-1296

Facility Description

The West Sacramento facility covers 66,000 square feet, 43,000 of which is devoted to laboratory areas where routine sample analyses for organic and inorganic constituents are performed. Work hours are 8:00 AM to 5:00 PM Monday through Friday. Samples may be received until 6:00 PM Monday through Friday, and 8:00 AM to 2:00 PM Saturdays. The laboratory supports all major Department of Defense programs, and possesses laboratory accreditations in fourteen states.

As innovators in research and technology, the West Sacramento laboratory leads in the application of state-of-the-art instrumentation to environmental problems. The laboratory performs analyses by Liquid Chromatography/Mass Spectrometry (LC/MS) for explosives residues, pesticides and specialty chemicals; Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) for low-level metals analyses; and High Resolution Gas Chromatography/High Resolution Mass Spectrometry for trace level detections of dioxins/furans, co-planar polychlorinated biphenyls, and polynuclear aromatic hydrocarbons in a variety of matrices. The facility also provides testing for nonvolatile components in support of air monitoring programs, including dioxins, metals, anions, and base/neutral organic compounds.

ATTACHMENT B

LABORATORY STANDARD OPERATING PROCEDURES WITH SUPPLEMENTAL SOP BINDER

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES, SW-846 METHOD 6010B AND EPA METHOD 200.7

(SUPERSEDES: REVISION 1)

Prepared by: Sevda Aleckson and Bill Nash

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Approved by: _____
Management,

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 3
2. SUMMARY OF METHOD 3
3. DEFINITIONS 4
4. INTERFERENCES 4
5. SAFETY 5
6. EQUIPMENT AND SUPPLIES 6
7. REAGENTS AND STANDARDS 7
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 8
9. QUALITY CONTROL 8
10. CALIBRATION AND STANDARDIZATION 15
11. PROCEDURE 15
12. DATA ANALYSIS AND CALCULATIONS 19
13. METHOD PERFORMANCE 21
14. POLLUTION PREVENTION 22
15. WASTE MANAGEMENT 22
16. REFERENCES 22
17. MISCELLANEOUS (TABLES, APPENDICES, ETC.) 23

LIST OF APPENDICES:

APPENDIX A - TABLES 27
APPENDIX B - QUANTERRA ICP DATA REVIEW CHECKLIST 38
APPENDIX C - CROSS REFERENCE OF TERMS USED IN METHODSAND IN SOP 40
APPENDIX D - MSA GUIDANCE 42
APPENDIX E - TROUBLESHOOTING GUIDE 44
APPENDIX F - CONTAMINATION CONTROL GUIDELINES 46
APPENDIX G - PREVENTATIVE MAINTENANCE 48

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7 . Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.5. State-specific requirements may take precedence over this SOP for drinking water sample analyses

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is

transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.

- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**:

sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

~~1.1.2.~~5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

~~1.1.3.~~5.3.3. The plasma emits strong UV light and is harmful to vision. **NOTE: AVOID looking directly at the plasma.**

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood or well ventilated area.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Argon gas supply, welding grade or equivalent.

6.4. Coolflow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom Quanterra multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix as listed in Section 11.10.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis. For samples that will be analyzed by Method 200.7 for compliance with Safe Drinking Water regulations, the samples must be held for a minimum of 16 hours prior to verifying the pH.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation .

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

~~1.1.~~9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

~~1.1.1.~~9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the Quanterra reporting limit. The CLP IDL procedure can be used for this method.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be considered valid. The result of the MDL determination must be below the Quanterra reporting limit (RL). MDL studies for the determination of metals in soil need not be performed; an appropriate soil MDL may be computed from the experimentally determined MDL for metals in aqueous solution.

9.1.3. Linear Range Verification (LR) - The linear range must be determined on an annual basis for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear range limit must be analyzed during a routine analytical run. For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.4. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a

computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.5. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response.

~~1.1.6.~~9.1.6. Rinse Time Determination - Rinse times must be determined annually. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies

must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the

laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, for method 6010B, a control limit of 80 - 120% (85-115% for 200.7) recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% (115% for 200.7) and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the report narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For both methods 200.7 and 6010B, control limits of 75 - 125% recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective

action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 or 11.12 for required run sequence).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is be a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See

Section 11.11 or 11.12 for required run sequence.) The CCB result must fall within +/- RL from zero. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence).

9.8. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom Quanterra multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.8.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.8.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.11 or 11.12 for required run sequence.)

9.8.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.8.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the Quanterra guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs> 10 µg/L must fall within the Quanterra guidelines of ± 1x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

- If the affected element was not required then the sample data can be accepted.

- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.

- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.

- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.10. Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC which may supersede this SOP requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understand the special project requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all

calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

~~1.2.~~11.2. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4. The use of an autosampler for all runs is strongly recommended.

11.5. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.6. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.8. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.9. The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful

in overcoming matrix interferences especially in high solids matrices. However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

11.10. The use of an internal standard is **required** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
CLP Aqueous	1	5
CLP Soil	5	2.5
SW846 3050	5	10
SW846 3005	2	5
SW846 3010	3	5

The following procedural guidelines must be followed when using an internal standard:

~~1.1.1.~~11.10.1. Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.10.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.10.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.10.4. The internal standard raw intensity counts must be printed on the raw data.

11.10.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.10.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.10.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

~~1.11.~~11.11. The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
ICSA
ICSAB
8 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B and 200.7 quality control criteria.

~~1.12.~~11.12. Additional quality control analyses are necessary for analysis under the Contract Laboratory Program (CLP). If these are included then CLP, 6010 and 200.7 samples can be included in the same sequence. Refer to CORP-MT-002 for details.

11.13. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.14. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.15. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.16. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix E provides guidance on performing MSA analyses.

~~1.17.~~11.17. Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

11.18. Nonconformance documentation shall be filed in the project file.

11.19. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

C4-155

~~1.2.~~12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

~~1.3.~~12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

~~1.4.~~12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

~~1.5.~~12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

~~1.6.~~12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

~~1.7.~~12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

~~1.8.~~12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

~~1.9.~~12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25 % and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% (15% for 200.7) of the true value or within the laboratory's historical acceptance limits.

13.4. Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed of according to the facility hazardous waste procedures and per the local, state, and federal regulations. The Environmental Health and Safety Director should be contacted, if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.2. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.3. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

16.4. CORP-MT-0002, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 & CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from both Methods 6010B and 200.7.

17.1.1.1. Quanterra laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

17.1.1.2. The alternate run sequence presented in Section 11.12 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol requirements.

17.1.1.3. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," Quanterra has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.4. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because Quanterra laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, Quanterra may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At Quanterra labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. Quanterra requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. Quanterra labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method section 9.3.4 specifies that "Analysis of the IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4." Quanterra uses a minimum of two exposures.

17.1.2.4. Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.5. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.6. Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.7 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDLIf not, repeat the analysis two or more times and average the results. If the average is not within three standard deviation of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.7 for a detailed description of the required corrective action procedures.

17.2. Modifications from previous SOP

Refer to revision 1 of this SOP.

~~1.3.~~17.3. Facility-Specific SOPs

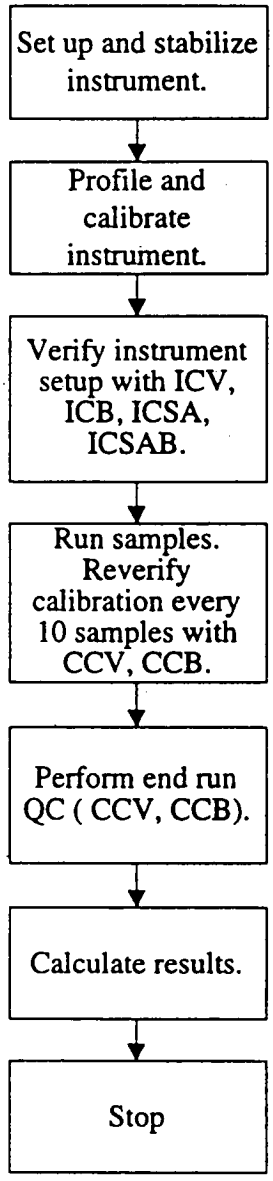
Each facility shall review and revise as appropriate this SOP to reflect any facility-specific requirementsIf no facility-specific amendments are required, the SOP can be adopted as is.

~~1.4.~~17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

ELEMENT	Symbol	CAS #	6010B analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Lithium	Li	7439-93-2	X	X	50	5
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Phosphorus	P	7723-14-0	X	X	300	30
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silicon	Si	7631-86-9		X	500	N/A
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-24-6	X		50	5
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5
Bismuth	Bi	7440-06-99	200	20
Zirconium	Zr	7440-06-77	100	10
Tungsten	W	7440-03-37	500	50
Tellurium	Te	1349-48-09	500	50
Thorium	Th	7440-02-91	500	50
Uranium	U	7440-06-11	500	50
Palladium	Pd	7440-00-53	100	10

NOTE: Analysis of all elements listed may not be available at all Quanterra facilities.

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Phosphorous	10000	10000
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000
Bismuth	1000	1000
Zirconium	1000	1000
Tellurium	1000	1000
Thorium	1000	1000
Uranium	1000	1000
Tungsten	1000	1000
Palladium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	5	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Lithium	10000	50	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Phosphorous	10000	300	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Strontium	10000	50	1000	5000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000
Boron	10000	200	1000	5000
Silicon	10000	500	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000
Bismuth	10000	200	1000	5000
Zirconium	10000	100	1000	5000
Tellurium	10000	500	1000	5000
Thorium	10000	500	1000	5000
Uranium	10000	500	1000	5000
Tungsten	10000	500	1000	5000
Palladium	10000	100	1000	5000

TABLE IVA. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom Quanterra solutions contain analytes common to all Quanterra facilities. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY*	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.8.3	See Section 9.8.3.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.8.2.

* See Sections 11.11 and 11.12 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Dilution Test	One per prep batch.	For samples > 50x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2). Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. (85-115% for 200.7) Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. I For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

QUANTERRA ICP DATA REVIEW CHECKLIST

Quanterra ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **6010B / 200.7: CORP-MT-0001 Rev 2**
CLP ILMO3.0/4.0 : CORP-MT-0002 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (6010B, CLP = 90 - 110%, 200.7 = 95 -105%[ICV])				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. CRI analyzed? (for CLP only)				
5. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Dilution Test done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND BY QUANTERRA

CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND QUANTERRA INC. SOP

EPA 200.7	SW6010B	Quanterra Inc. SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

C4-178a

APPENDIX D

MSA GUIDANCE

C4-178b

APPENDIX E. TROUBLESHOOTING GUIDE

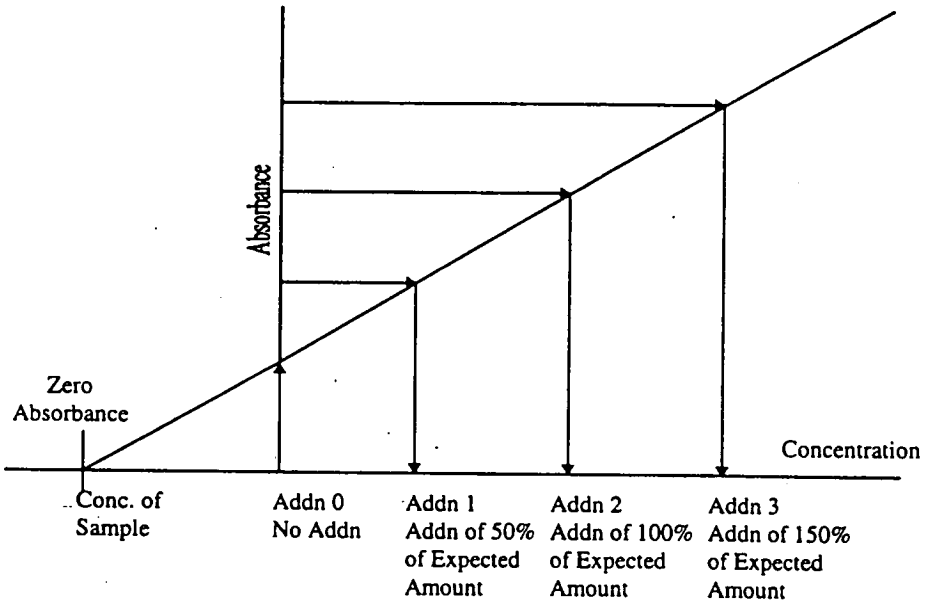
Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing and pump windings Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check Cu/Mn ratio-should be 30% of value at date that IECs were performed Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
Weekly	Apply silicon spray on autosampler tracks Check water level in coolflow
Monthly	Clean air filters on back of power unit to remove dust Check D mirror for air instruments
Bi-yearly	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

Quanterra
Environmental Services

SOP No. SAC-LC-0001
Revision No. 5.0
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Page 1 of 63

DETERMINATION OF NITROAROMATICS, NITRAMINES, AND SPECIALTY EXPLOSIVES IN WATER AND SOIL BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/ULTRAVIOLET DETECTOR (HPLC/UV) AND LIQUID CHROMATOGRAPHY/THERMOSPRAY/MASS SPECTROMETRY (LC/TSP/MS)

SUPERSEDES: Revision 4.0; August 3, 1995

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1. SCOPE AND APPLICATION

1.1 This method is suitable for the extraction and analysis of explosive residues (8330 components) listed below in water by high level and low level methods and in soil and sediment by High Performance Liquid Chromatography with Ultraviolet Detector (HPLC/UV) and with Triple-Stage Quadrapole Mass Spectrometer (LC/TSP/MS).

Compound	CAS No
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX, Octogen)	2691-41-0
1,3,5-trinitrobenzene	99-35-4
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX, Hexogen)	121-82-4
1,3-dinitrobenzene	99-65-0
Nitrobenzene	98-95-3
2,4,6-trinitrotoluene (TNT)	118-96-7
Methyl-2,4,6-trinitrophenylnitramine (TETRYL)	479-45-8
2,4-dinitrotoluene	121-14-2
2,6-dinitrotoluene	606-20-2
2-Amino-4,6-Dinitrotoluene	35572-78-2
4-Amino-2,6-Dinitrotoluene	1946-51-0
2-nitrotoluene (o-nitrotoluene)	88-72-2
4-nitrotoluene (p-nitrotoluene)	99-99-0
3-nitrotoluene (m-nitrotoluene)	99-08-1

1.1.1 This method is NOT applicable for high concentrations of explosives in soil or waste samples. Method 8510 should be used to determine whether high concentrations of explosives are present in soil or waste samples.

1.1.2 For HPLC/UV, the standard range for most of the 8330 compounds is from 5.0 ng/mL to 1000 ng/mL, except for 1,3-dinitrobenzene and 2,4-dinitrotoluene which are from 2.5 ng/mL to 1000 ng/mL, in 25/75 0.1% acetic acid in acetonitrile/water,

1.2 This method is applicable for the extraction and analysis of 8330 compounds previously listed above and the following specialty explosives listed below by LC/TSP/MS.

Compound	CAS No
Nitroglycerine	55-63-0
Picric Acid (2,4,6-Trinitrophenol)	88-89-1
PETN	78-11-5

1.2.1 For LC/TSP/MS, the standard range is from 10 ng/mL to 1000 ng/mL for 8330 components, nitroglycerin, and PETN, and from 20 ng/mL to 2000 ng/mL for picric acid in 20/80 acetonitrile/0.2M ammonium acetate containing 0.1% acetic acid.

1.2.2 Aqueous samples for 8330 and specialty explosives can be extracted according to the low level or high level method, except for picric acid and nitroguanidine which are performed by the high level direct aqueous injection (DAI) method.

1.2.3 Soil samples for 8330 and specialty explosives can be extracted with 0.1% acetic acid in acetonitrile according to 8330 soil method except for picric acid which results in poor recovery.

1.2.4 Excellent recoveries for picric acid can be obtained from soil by extracting with 0.1% hydrochloric acid in 10% acetonitrile/water, however, this is not suitable for 2-amino-4,6-dinitrotoluene and 4-amino-2,6-dinitrotoluene which degrade rapidly.

1.2.5 With the addition of acetic acid in the extracting solvent and in the standard solutions, TETRYL has been stable for months without affecting the other nitroaromatics and nitramines.

1.3 This method is also suitable for the extraction and analysis of nitroguanidine in water and in soil by HPLC/UV.

Compound	CAS No
Nitroguanidine	556-88-7

1.3.1 The standard range for nitroguanidine by HPLC/UV is from 0.05 ug/mL to 2.0 ug/mL in 80/20 acetonitrile/water.

1.3.2 Nitroguanidine is not extractable from water, but water samples are diluted with acetonitrile and analyzed by DAI on the HPLC/UV.

1.3.3 Nitroguanidine is extractable from soil as per 8330 soil with 0.1% acetic acid in acetonitrile.

2. SUMMARY OF METHOD

2.1 Aqueous samples are either extracted and analyzed by the high level or the low level method, and analyzed by HPLC/UV or by LC/TSP/MS.

2.2 Aqueous samples for the high level method can be performed by DAI, where the samples must be analyzed within 7 days of sampling, or by the salting-out method or by solid phase extraction (SPE) where the extraction is performed within 7 days of sampling and analyzed within 40 days of extraction.

2.2.1 There is no extraction of aqueous samples for nitroguanidine except by DAI; samples are aliquotted, diluted with acetonitrile to extend the analytical hold time, filtered and analyzed by HPLC/UV.

2.2.2 Picric acid is performed by the high level DAI method; samples are aliquotted, diluted in ammonium acetate, filtered, internal standards are added, and the sample is analyzed by LC/TSP/MS.

2.2.3 Most other components can be performed by the high level or the low level salting-out method or SPE method.

2.2.4 The high level salting-out method and the high level SPE method are similar to the low level methods where the volume of the samples and reagents are downscaled approximately 10 to 1 with higher fortification levels.

2.3 Aqueous samples for the low level salting-out method are performed by saturating the water sample with sodium chloride and extracting with acetonitrile several times. The volume of the acetonitrile extracts are reduced by re-extracting with salt water, and adjusted to 5.0 mL. The extracts are diluted 1:3 before analysis. For HPLC/UV the extracts are diluted in water, and for LC/TSP/MS the extracts are diluted in 0.2 M ammonium acetate and internal standards are added.

2.4 Aqueous samples for the low level SPE method are performed by passing water samples through Porapak RDX cartridges, where the components are absorbed to and eluted from with 0.1% acetic acid in acetonitrile. The eluants are diluted with water for HPLC/UV or with 0.2 M ammonium acetate and internal standards added for LC/TSP/MS.

2.5 This method is NOT applicable for high concentrations of explosives in soil or waste samples.

2.6 Soil and sediment samples that appear to be non-homogeneous and are not suspicious are air dried, ground to a finer texture, and sieved through 30 mesh. A subsample is extracted for 8330, nitroguanidine, nitroglycerine, and PETN by using 0.1% acetic acid in acetonitrile in a cooled ultrasonic water bath for 18 hours, aliquotted, treated with calcium chloride solution (except for LC/MS), filtered, and analyzed by HPLC/UV or by LC/TSP/MS.

2.6.1 Acetic acid at 0.1% in acetonitrile improves the stability of TETRYL, but gives poor recoveries for picric acid in soil.

2.6.2 Picric acid is extracted from soil with 0.1% hydrochloric acid in 10% acetonitrile/water in a cooled ultrasonic water bath for 18 hours, filtered, aliquotted, internal standards added, and analyzed by LC/TSP/MS.

2.7 The 8330 extracts for HPLC/UV are analyzed on a C-8 reverse phase column with UV detection at 250 nm and confirmed on a Cyano (CN) column using reverse phase conditions and UV detection at 250 nm, or on a C-8 reverse phase column by LC/TSP/MS.

2.8 The extracts may be analyzed and confirmed by LC/TSP/MS using a C-8 reverse phase column.

3. DEFINITIONS

3.1 Interference - an obstruction or contamination at a similar retention time as the compound of interest.

3.2 Method Blank (MB) - A single control sample using similar controlled matrix in a set or batch for monitoring and to demonstrate that the reagents and solvents used are free from method interferences.

3.3 Laboratory Control Samples (LCS) - A single control sample using similar controlled matrix and fortified with target analytes.

3.4 Duplicate Control Samples (DCS) - A LCS performed in duplicate and fortified with target analytes.

3.5 Internal Standard - A compound either may be similar or not similar to the target analytes and may be isotopically labelled, is added to the final extracts to aid in determining that a valid injection has been made and may be used in the quantitation.

3.6 Matrix Spike (MS) - A single sample in a set or batch either selected at random or selectively choosen and fortified with target analytes.

3.7 Matrix Spike Duplicate (MSD) - A matrix spike performed in duplicate and fortified with target analytes.

3.8 Surrogate - A compound similar to the target analytes, may be isotopically labelled, to demonstrate the efficiency of the extraction on the sample.

3.9 Low Level method - Aqueous samples to be extracted according to the low level method for lower reporting limits of approximately 0.1 to 1.0 ng/mL (ppb).

3.10 High Level method - Aqueous samples to be extracted according to the high level method for higher reporting limits of approximately 4.0 to 14 ng/mL (ppb). NOTE: Explosive components are not very soluble in water, thus, there is no danger of possible re-crystalization and becoming dangerously explosive.

4. INTERFERENCES

4.1 Glassware must be cleaned and dried, free from interference.

4.2 The solvents and reagents used in the extraction and analysis of nitroaromatics, nitramines, and specialty explosives must be free of interference.

5. SAFETY

Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.1 Eye protection that satisfies ANSIZ87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. {VITON gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]}

5.2 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include methylene chloride (used primarily in the rinsing of glassware).

Chemicals known to be flammable are acetone, acetonitrile, hexane, methanol, n-propanol, and tetrahydrofuran.

Tetrahydrofuran is known to be explosive as it forms explosive peroxides on prolonged storage.

Explosive standards in this method are secondary explosives. As neats, they are by nature unstable materials and they must be stored according to manufacturer's directions, preferrably wet; However, if the standards are not purchased as neats but as solutions at low concentrations in organic solvents, safety hazards is minimized as compare to neats.

Chemicals known to be corrosive are acetic acid and hydrochloric acid.

Hearing protection may be recommended when ultasonic digestion is carried out.

5.3 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.4 The preparation of standards and reagents (and glassware cleaning procedures that involve solvents such as methylene chloride) will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

5.6 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, from this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of Material Safety Data Sheets [MSDS] for chemicals used or encountered in this procedure should be and are made available to all personnel involved in the chemical analysis.

5.7 Each chemical compound must be treated as a potential health hazard, especially when the components are considered extremely explosive and should be kept wet in small quantities. All the compounds in this method are either used in the manufacturing of explosives or are the degradation products of compounds used in the manufacturing of explosives. Treat each compounds as if it were extremely explosive when handling and preparing stock solutions for calibration.

5.8 Soil samples having as high as 2% TNT can be safely ground. Samples containing higher levels should not be ground. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

5.8.1 Soil samples may be screened or tested before grinding if they are suspected to contain high levels of explosives using the TNT/RDX Soil Extraction Pac. Follow the manufacturer's instructions.

6. EQUIPMENT AND SUPPLIES

6.1 Balances - Top loading, capable of accurately weighing to the nearest 0.01 gram. Analytical, capable of accurately weighing to the nearest 0.0001 gram.

6.2 Centrifuge.

6.3 Filter assembly, 25-mm, 0.45 um pore size PTFE filters, such as Gelman Acrodisc #4219.

6.4 Liquid chromatograph

6.4.1 HPLC/UV - High Performance Liquid Chromatography with an Ultraviolet Detector. An analytical system complete with sample injection loop, analytical columns, a UV detector, and data system. A guard column is not used. See section 12.7, 12.8, and 12.9 for the appropriate HPLC conditions.

6.4.2 LC/TSP/MS - Liquid Chromatography/Thermospray/Mass Spectrometer. An analytical system complete with sample injection loop, analytical columns, a triple-stage quadrapole mass spectrometer, and data system. See section 12.10 for LC/TSP/MS conditions.

6.5 Mesh sieve and pan, brass or stainless steel, mesh size # 30.

6.6 Mortar and pestle, ceramic.

6.7 Recirculator cooler.

6.8 Spatula, stainless steel, or equivalent.

6.9 Stirring bars, magnetic.

6.10 Stirring plate, magnetic.

6.11 Syringe, 3-cc and 10-cc disposable.

6.12 Temperature controlled ultrasonic water bath.

6.13 Test tubes, 8-mL and 16-mL, glass with teflon-lined screwcap.

6.14 Vials, 40-mL, glass with teflon-lined screwcap.

6.15 Volumetric flask, 100-mL and 1000-mL.

7. REAGENTS AND SOLVENTS

All preparations of reagents and solutions are entered in the Reagent Prep Notebook.

7.1 Acetic acid, Glacial, Reagent grade.

7.1.1 Acetic acid in acetonitrile, 0.1% v/v.

7.2 Acetone, pesticide quality.

7.3 Acetonitrile, HPLC grade or equivalent.

7.4 Ammonium acetate, HPLC grade or equivalent.

7.4.1 Ammonium acetate, 0.2 M and 4 M (aqueous).

7.5 Calcium chloride, $CaCl_2$, aqueous solution; 1.3 grams/liter in distilled water.

7.6 Hexane, pesticide quality.

7.7 Hydrochloric acid, concentrated, reagent grade.

7.7.1 Hydrochloric acid in 10% acetonitrile/water, 0.1% v/v.

7.8 Methanol, HPLC grade or equivalent.

7.9 Methylene chloride, pesticide quality.

7.10 n-Propanol, HPLC grade or equivalent.

7.11 RDX/TNT Soil Extraction Pac, D TECH product, Accustandard catalog # EM-TK-1001S-1 or equivalent. Each kit contains materials to perform four tests.

7.12 Sodium chloride, reagent grade.

7.12.1 Salt water; 325 grams of sodium chloride in 1 liter of HPLC grade water (distilled water may be used for 8330 by LC/TSP/MS).

7.13 Tetrahydrofuran (THF), HPLC grade or equivalent.

7.14 Water, distilled.

7.15 Water, HPLC grade or equivalent.

8. STANDARDS

8.1 Stock standards and solutions may be prepared from standard materials or purchased as certified solutions. All standard preparations must be entered in the standards prep log (see Appendix 1).

A prepared mix solution of the 8330 components at 1.0 mg/mL in acetonitrile/methanol (1:1) is used for the preparation of fortification and analytical solutions. They are obtained from EM Science, Accustandard, Ultra Scientific, or other reputable source. The mix solutions from Accustandard and Ultra Scientific contained 14 components at 1.0 mg/mL, and expires in approximately one year if properly stored. Once the ampule is broken and the unused portion transferred to an amber vial, sealed with teflon-lined screwcap, and stored in the freezer, this solution is good for up to 30 days.

8.1.1 Prepare a 50 ug/mL mix solution in 0.1% acetic acid in acetonitrile from the primary mix standard above. Replace the solution after 30 days.

8.1.2 Prepare a 5.0 ug/mL mix solution from the 50 ug/mL mix solution from above in 0.1% acetic acid in acetonitrile. Replace the solutions after approximately 2 weeks.

8.1.3 Both solutions above are stored in the freezer in the dark and at -10 °C or less. These solutions are also used for fortification of samples.

8.2 Standards must be reviewd for expiration dates at least monthly. All expired standards must be rotated out of the laboratory to the HazWaste storage area for disposal per the CHP.

8.3 Surrogate for HPLC/UV.

8.3.1 Several chemicals listed below can be used as surrogate.

1-Chloro-3-nitrobenzene (CAS No. 121-73-3)

2,4-Dinitrofluorobenzene (CAS No. 70-34-8)

NOTE: Both compounds resolve from the 8330 analytes on the C8 analytical column, but only 2,4-dinitrofluorobenzene resolves from the 8330 analytes on the cyano confirmation column. Depending on the manufacturer and lot of the cyano column, 1-chloro-3-nitrobenzene co-elutes with RDX or with 3-nitrotoluene.

8.3.2 Prepare a stock solution from neat (obtained from Aldrich) at approximately 1.0 mg/mL in acetonitrile. Store the stock solution in the refrigerator and replace the stock after 1 year.

8.3.3 Prepare the surrogate solution from stock at 50 ug/mL in acetonitrile. Store the solution in the freezer and replace every six months.

8.4 Surrogate for LC/TSP/MS; d_5-nitrobenzene (CAS No. 4165-60-0)

8.4.1 Prepare a stock solution d_5-nitrobenzene from neat (obtained from CIL) at approximately 1.0 mg/mL in acetonitrile. Store the stock solution in the refrigerator and replace the solution after 1 year.

8.4.2 Prepare the surrogate solution from stock at 50 ug/mL in acetonitrile. Store the solution in the freezer and replace every six months.

8.5 Analytical Curve for 8330 HPLC/UV.

8.5.1 The analytical curve is prepared from the 5.0 ug/mL 8330 mix solution and the 50 ug/mL surrogate solution at the following concentrations in 25:75 0.1% Acetic acid in acetonitrile:HPLC grade water.

LEVEL:	T1	1	2	3	5	6	7	
8330 mix	2.5	5.0	10	25	100	500	1000	ng/mL
Surrogate	5.0	25	50	100	150	--	--	

Level T1 standard is the lowest level used for 1,3-dinitrobenzene, 2,4-dinitrotoluene, and the surrogate whereas the others used level 1 standard in the initial calibration for the HPLC/UV.

Store the solutions in the refrigerator and replace every 45 days. TETRYL is stable when properly stored with acetic acid.

8.5.2 A continuing calibration standard is prepared from the 5.0 ug/mL 8330 mix solution and the 5.0 ug/mL surrogate solution at 25 ng/mL with the surrogate at 100 ug/mL in 25:75 of 0.1% acetic acid in acetonitrile:HPLC grade water. Store the solution in the refrigerator and replace every 45 days.

8.6 Nitroguanidine is prepared from neat (obtained from Aldrich or Chem Service) at approximately 1.0 mg/mL in acetonitrile. Store the stock solution in the refrigerator and replace after 1 year.

8.6.1 Prepare nitroguanidine at 50 ug/mL and 5.0 ug/mL from the 1.0 mg/mL stock in acetonitrile. Store the solutions in the refrigerator and replace after six months. These solutions can also be used for fortification of samples.

8.6.2 Prepare the analytical curve for HPLC/UV from the 50 ug/mL standard at the following concentrations in 80:20 acetonitrile:water. Store the solutions in the refrigerator and replace after three months.

LEVEL:	1	2	3	4	5
NGu	0.05	0.10	0.20	1.0	2.0 ug/mL

8.7 SPECIALTY EXPLOSIVES (SPEX) - Nitroglycerine, picric acid, and PETN are received as solutions at 0.10 mg/mL or at 1.0 mg/mL in ethanol, methanol, or acetonitrile (obtained from EM Science, Accustandard, Ultra Scientific, or other reputable source).

8.7.1 A mixture of nitroglycerine, picric acid, and PETN (NGl/PA/PETN) is prepared at 20/20/10 ug/mL in acetonitrile. Store the solution in the freezer and replace the solution after 6 months. This solution can also be used for fortification of samples.

8.8 Internal Standards for LC/TSP/MS;

d_3-2,6-Dinitrotoluene (CAS No. 606-20-2) and d_4-1,3-dinitrobenzene (CAS No. 99-65-0)

8.8.1 Prepare individual stock solutions of d_3-2,6-dinitrotoluene and d_4-1,3-dinitrobenzene from neats (obtained from CIL) at approximately 1.0 mg/mL in acetonitrile. Store the stock solutions in the refrigerator and replace after 1 year.

8.8.2 Prepare an internal standard mix of d_3-2,6-dinitrotoluene and d_4-1,3-dinitrobenzene solution at 10 ug/mL in acetonitrile. Store the solution in the refrigerator and replace after 6 months.

8.9 Analytical Curve for LC/TSP/MS.

8.9.1 The 8330 and specialty explosives (SPEX) analytical curve is prepared at the following concentrations listed below in 2:8 acetontrile:0.2M ammonium acetate containing 0.1% acetic acid.

LEVEL:	1	2	3	4	5
8330 mix	10	50	100	500	1000 ng/mL
NGl	20	100	200	1000	2000
PA	20	100	200	1000	2000
PETN	10	50	100	500	1000
d_5-NB	50	100	150	200	250
d_3-DNT	50	50	50	50	50
d_4-DNB	50	50	50	50	50

where the 8330 mix is the 14 components, NGl is nitroglycerin, PA is picric acid, d_5-NB is the surrogate d_5-nitrobenzene, and d_3-DNT and d_4-DNB are the internal standards d_3-2,6-dinitrotoluene and d_4-1,3-dinitrobenzene.

8.9.2 Store the solutions in the refrigerator and replace after six months. TETRYL is stable when properly stored with 0.1% acetic acid.

8.10 Reference Standards

8.10.1 Prepare reference standards to verify the quality of the fortification solutions and analytical standards above.

8.10.2 Reference standard is any standard solution made from a source other than the stock standard. Reference standard may be from the EPA, the manufacturer, or from another reliable source.

8.10.3 If the secondary source is not available, a separate intermediate stock solution will be made from the same neat by another chemist or from a neat with a different lot number.

9. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, AND HOLDING TIMES

9.1 Aqueous samples are collected in amber glass bottles with teflon-lined caps and iced or refrigerated at 2 - 6 °C from the time of collection until analysis.

9.2 Aqueous samples for the high level method must be aliquotted, filtered, and diluted within 7 days of sampling or extracted within 7 days and analyzed within 40 days from extraction.

9.2.1 If aqueous samples are not diluted in organic solvent, then the samples must be analyzed by DAI within 7 days of sampling.

9.3 Soil samples are collected in glass jars with teflon-lined caps and iced or refrigerated at 2 to 6 °C and covered from sunlight from time of collection until extraction.

9.4 Soil samples for 8330 and specialty explosives are to be air dried, finely ground and sifted, and extracted within 14 days of sampling and analyzed within 40 days of extraction.

9.4.1 Soil and sediment samples should be air dried until there is no visible appearance of moisture at room temperature or cooler after collection. Successful grinding and sifting verifies dryness. While it is possible to analyze wet soil samples, it is much more difficult to obtain a homogeneous subsample on a wet sample.

9.4.2 If wet soil samples are to be analyzed, a moisture determination must be made on a separate subsample (refer to SOP #LM-CAL-3055, "Determination of Percent Moisture (or Percent Solids)").

10. QUALITY CONTROL

10.1 A set or batch is a group of samples processed at the same time and by the same technique.

10.2 Any method blanks (MB) and control samples should be subjected to the same analytical procedures as those used on the actual samples at the same time.

10.2.1 A minimum of one method blank per batch or every twenty samples is required, whichever is more frequent.

The MB for aqueous samples consists of distilled water and for soil samples consists of Ottawa sand taken through the sample prep with the samples.

10.3 Laboratory Control Samples (LCS) are required and must be prepared for every 20 samples of each matrix, where MS/MSD are performed in the batch.

10.3.1 Duplicate Control Samples (DCS) are required with every batch where MS/MSD are not included.

LCS and DCS for water samples consists of distilled water and for soil samples consists of Ottawa sand fortified and taken through the sample prep with the samples.

10.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are required per batch.

10.4.1 The MS/MSD consists of two equal aliquots of a specified sample and are fortified at the same concentrations as the LCS or at client's request.

10.5 The surrogate is added to all samples in the batch, including the MB, LCS, DCS, MS, MSD, and Duplicates.

11. CALIBRATION AND STANDARDIZATION

11.1. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

11.2 Initial Calibration - A minimum of five analytical standards of different analyte concentrations are used to generate the curve.

11.2.1 On the HPLC/UV for 8330 and for nitroguanidine analysis, the initial calibration is injected once to obtain the peak height for each analyte at each concentration, and the response factor is calculated using the following formula (from VG Multichrom data system):

$$R_f = (\text{peak height})/(\text{analyte concentration})$$

11.2.2 The average response factor (R_fa) and the percent relative standard deviation (% RSD) is determined for each analyte. The average response factor is the summation of the R_f/n where n is the number of data points (n = 5 for five levels).

11.2.3 The initial calibration is valid for each analyte when the % RSD is equal to or less than 20%; linearity can be assumed and the average response factor can be used in place of a calibration curve (on the HPLC/UV).

11.2.3.1 If the % RSD is greater than 20 %, then the cause must be determined to bring the system back in control before the samples are analyzed, and the system must be recalibrated.

11.2.4 On the LC/TSP/MS for 8330 and specialty explosives, the initial calibration curve is generally injected at the beginning of the batch to obtain the area counts for each analyte at each concentration, and the response factor is calculated using the following formula (on Finnigan DEC Station 5000 data system):

$$R_f = \frac{C_s}{R_s} \times \frac{R_{is}}{C_{is}}$$

where C_s is the concentration of the standard in ng/mL,
R_s is the area count of the standard
C_{is} is the concentration of the internal standard,
R_{is} is the area count of the internal standard.

11.2.5 The average response factor (R_fa) and the percent relative standard deviation (% RSD) is determined for each analyte, and the initial calibration is valid for each analyte when the % RSD is equal to or less than 30%; linearity can be assumed and the average response factor can be used in place of a calibration curve (on the LC/TSP/MS).

11.2.5.1 If the % RSD is greater than 30 %, then the cause must be determined to bring the system back in control before samples are analyzed, and the system must be recalibrated.

11.2.6 All units used in the calculations must be consistently uniform, such as concentration in ng/mL and response in area or height counts.

11.3 Calibration Criteria - On the HPLC/UV, at the beginning of the batch, the continuing calibration standard is injected singly to determine the concentration for each analyte using the response factors from the initial calibration. The value calculated is compared to the expected value and should be equal to or less than 15% to be valid, and the samples may be analyzed.

11.3.1 The continuing calibration standard is the midpoint standard, and throughout the run it is the same.

11.3.2 If the continuing calibration standard is out of control, another injection of the continuing calibration standard may be injected singly to determine if it is back in control. If it's still out of control, then either the midpoint standard has degraded or the column has degraded and a new initial calibration is needed.

11.3.3 The initial calibraton curve is valid if the continuing calibration standard injected at the beginning of the analytical batch holds within the 15% criteria.

11.3.3.1 Minimally, if the 15% criteria is met, a five point curve is performed each year. However, the typical life of the column for 8330 by HPLC/UV is approximately two months of continuous use.

11.3.4 Calibration Criteria on the LC/TSP/MS - At the beginning of the batch, the initial calibration standards are injected to obtain the average respose factor for each analyte and the % RSD is calculated. If the % RSD is equal to or less than 25% then the initial calibration is valid and the analytical batch is analyzed.

11.3.5 A continuing calibration standard is injected at the beginning and singly for every 10 samples or less, and at the end of the batch for HPLC/UV and LC/TSP/MS.

11.3.6 The single injections of the continuing calibration standard on the HPLC/UV must be within 15% of the expected value based on the $R_f a$ from the initial calibration to be valid.

11.3.7 On the LC/TSP/MS the continuing calibration standard must be within 25% of the expected value from the initial calibration.

12. PROCEDURE

One time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

NOTES: If Direct Aqueous Injection can not be performed within the 7 day holding time for the high level 8330 and specialty explosives then the hold times can be extended by performing the salting-out method or the SPE method, however, the high level salting-out method and the SPE method are not applicable for nitroguanidine and picric acid.

The SPE method for 8330 has been approved by the USACOE and may be used in place of the salting-out method.

12.1 **HIGH LEVEL method by DAI;** for nitroguanidine (NGu), 8330, and Specialty Explosives (SPEX).

12.1.1 Label the sample ID with the appropriate suffix '8330-H-A', 'SPEX-H-A', 8330/SPEX-H-A, and 'NGu-A'.

12.1.2 Pipet 10 mL water sample into a 16-mL screwcap test tube.

For control, use distilled water or HPLC grade water.

12.1.3 Add the surrogate to the samples, including the method blank, DCS, LCS, and MS/MSD.

12.1.3.1 Surrogate is not applicable for nitroguanidine. The surrogates used for HPLC/UV and LC/TSP/MS do not inferfere with the analysis.

12.1.3.2 For 8330 by HPLC/UV, add 20 uL of the 50 ug/mL surrogate to the 10 mL water samples to yield 100 ppb (DO NOT ADD d_5-Nitrobenzene as it interferes with nitrobenzene on HPLC/UV).

12.1.3.3 For 8330, 8330/SPEX, or SPEX by LC/TSP/MS, add 40 uL of the 50 ug/mL d_5-Nitrobenzene standard to the 10 ml water samples to yield 200 ppb.

12.1.4 Add the spiking solution if applicable.

12.1.4.1 For nitroguanidine, add 160 uL of the 50 ug/mL nitroguanidine solution to the 10 mL water samples to yield 800 ppb.

12.1.4.2 For 8330 by HPLC/UV, add 100 uL of the 5.0 ug/mL 8330 mix solution to the 10 mL water samples to yield 50 ppb.

12.1.4.3 For SPEX by LC/TSP/MS, add 50 uL of the 20/20/10 ug/mL Nitroglycerine/Picric Acid/PETN solution to the 10 mL water samples to yield 100/100/50 ppb.

12.1.4.4 For 8330/SPEX by LC/TSP/MS, add both standards below to 10 mL water sample: Add 100 uL of the 5.0 ug/mL 8330-mix to yield 50 ppb, and 50 uL of the 20/20/10 ug/mL NGl/PA/PETN mix to yield 100/100/50 ppb.

12.1.5 Mix the contents well, and filter the water sample, the method blank, LCS/DCS, and MS/MSD.

12.1.5.1 Transfer the water sample into a 10-cc disposable syringe fitted with a 25-mm PTFE 0.45 um filter on the syringe tip and press the plunger. Collect the sample into another 16-mL screwcap test tube.

12.1.6 For 8330 by HPLC/UV, the extracts are ready for analysis. The final sample concentration is 10mL/10mL. If the extracts are to be split for another test, then aliquot the required amount of the 10/10 into a separate 8-mL test tubes for other test.

12.1.6.1 Store the 8330-H-A extracts in the freezer until ready for analysis by HPLC/UV; This analysis must be completed within 7 days of sampling.

12.1.7 For Nitroguanidine (NGu-A), aliquot 1.0 mL of the 10/10 water sample into an 8-mL test tube and dilute with acetonitrile to final volume of 4.0 mL (a 1:3 dilution). Mix the contents well.

12.1.7.1 Store the NGu-A extracts in the freezer until ready for analysis by HPLC/UV. The final concentration is 1.0 mL/4.0 mL, and the analysis is to be completed within 40 days from dilution with acetonitrile.

12.1.8 For 8330 by LC/TSP/MS and Specialty Explosives (SPEX-A), aliquot 2.0 mL of the 10/10 water sample into an 8-mL test tube. Add 100 uL of 4 M ammonium acetate, 10 uL of internal standard mix of d_3-2,6-DNT and d_4-1,3-DNB at 50 ng/mL, and mix the contents well. The final sample concentration is 2.0mL/2.1mL, except for the LCS/DCS and MS/MSD is 1.9mL/2.1mL.

12.1.8.1 Store the extracts in the freezer until ready for analysis by LC/TSP/MS.

12.2 **HIGH LEVEL** salting-out method for 8330 and specialty explosives.

This method is not applicable for nitroguanidine or picric acid - use DAI.

12.2.1 Label the sample tape with the appropriate suffix '8330-H-A' or '8330/SPEX-H-A'.

12.2.2 Measure 77.0 mL of water sample by weight into a specialized 100-mL volumetric flask.

12.2.2.1 For control, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.2.3 Add surrogate to each sample, MB, LCS/DCS, and MS/MSD.

12.2.3.1 For 8330 by HPLC/UV, add 100 uL of the 50 ug/mL surrogate solution to the 77 mL water samples to yield 65 ppb.

12.2.3.2 For 8330/SPEX by LC/TSP/MS, add 200 uL of the 50 ug/mL d_5-Nitrobenzene solution to the 77 mL water samples to yield 130 ppb.

12.2.4 If applicable, fortify the LCS/DCS, and MS/MSD with the appropriate spiking standards.

12.2.4.1 For 8330-HPLC/UV, add 77 uL of the 50 ug/mL 8330 mix standard to the 77.0 mL water samples to yield 50 ppb.

12.2.4.2 For 8330/SPEX by LC/TSP/MS, add 77 uL of the 50 ug/mL 8330 mix and 0.40 mL of the 20/20/10 ug/mL NG1/PA/PETN mix to yield 50 ppb for 8330 mix and 100/100/50 ppb for SPEX.

12.2.5 Add 25.1 grams of sodium chloride (± 0.2 grams) to each water sample in the specialized volumetric flasks, including the control and spikes. Add the stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt dissolves.

12.2.6 Add 16 mL of acetonitrile while the solution is being stirred and stir for approximately 5 minutes at high speed. An efficient stirring is observed when the acetonitrile layer is vortexing in the water sample.

12.2.7 After stirring for 5 minutes, allow the phases to separate to determine if any acetonitrile is recoverable. Occassionally there is little or no acetonitrile layer visible but the sample is not lost. Add an extra 1 to 3 mL of acetonitrile, and stir the solution again.

12.2.8 After stirring for 5 minutes, check again to determine if acetonitrile is recoverable if extra volume had been added.

12.2.9 If there is sufficient volume of acetonitrile (2 to 4 mL), continue to stir for an additional 15 minutes (total time approximately 20 minutes).

12.2.10 When the stirring is completed, turn off stirrer and allow the phases to separate for a minimum of 10 minutes.

12.2.11 Carefully transfer the upper acetonitrile phase to an 8-mL test tube with a pasteur pipet.

12.2.12 Extract the aqueous sample once more with another 1 to 2 mL of acetonitrile (but do not exceed a total of 5 mL when combined), stir at high speed for 15 to 20 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract.

12.2.13 Carefully adjust the final volume to 5.0 mL with acetonitrile - NO nitrogen blowdown permitted.

12.2.14 Add 5 uL of concentrated acetic acid to each to stablize TETRYL, and mix the contents well.

12.2.15 Filter the extract through a 25-mm PTFE 0.45 um filter assembly using a 10-cc disposable syringe into another 8-mL test tube.

12.2.16 The acetonitrile extracts are stored in the freezer. A dilution of 1/10 is required for analysis to obtain a final concentration of 77mL/50mL - see below.

12.2.16.1 For HPLC/UV, dilute 0.4 mL of the extract with 3.6 mL distilled water in an 4-mL vial when ready for analysis.

12.2.16.2 For LC/TSP/MS, dilute 0.4 mL of the extract with 3.6 mL distilled water in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix well. Store the extracts in the freezer until ready for analysis by LC/TSP/MS.

12.3 HIGH LEVEL SPE method - EXTRACTION ON THE ZYMARK AUTOTRACE WORKSTATION

12.3.1 Label the sample ID with the appropriate suffix '8330-H-A'.

12.3.2 QC's setup (MB, DCS, LCS, and MS/MSD) for Zymark AutoTrace SPE

12.3.2.1 For MB, DCS, and LCS, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.3.2.2 If requested, control water supplied by the client may be used for MS/MSD. See section 12.3.4.1 below if sample contains sediments or suspended particulates.

12.3.2.3 Measure 100 mL of water sample by weight into an 8-oz glass bottle for QC's.

12.3.3 If applicable, fortify the DCS, LCS, and MS/MSD.

12.3.3.1 For 8330 by HPLC/UV, add 100 uL of the 50 ug/mL 8330 mix to the 100 mL water samples to yield 50 ppb.

12.3.3.2 For 8330/SPEX by LC/TSP/MS, add 100 uL of the 50 ug/mL 8330 mix to yield 50 ppb and 500 uL of the 20/20/10 ug/mL NGl/PA/PETN mix to yield 100/100/50 ppb in the 100 mL water samples.

12.3.4 Measure 100 ml of aqueous samples by weight into glass bottles in order to be able to add the surrogate to the samples.

12.3.4.1 If the water sample contains sediments or suspended particles, separate the particulates by centrifuging, filtering, or by decanting into a cleaned 1-L amber glass bottle.

12.3.5 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.3.5.1 For 8330 by HPLC/UV, add 100 uL of the 50 ug/mL surrogate solution to the 100 mL water samples to yield 50 ppb.

12.3.5.2 For 8330 and 8330/SPEX by LC/TSP/MS, add 250 uL of the 50 ug/mL d_5-Nitrobenzene standard to the 100 mL water samples to yield 125 ppb.

12.3.6 Weigh and record the initial mass of each sample bottle, including the QC's (with the screw caps on).

12.3.7 Samples ready for extraction by the Zymark Autotrace SPE.

12.3.8 The program for the Autotrace SPE workstation must be set up using the PC computer with the following parameters on a 3.5" disk.

Use the disk - 8330-A, and edit the sample volume to 80 mL.

12.3.9 Below is an overview of the program. Whenever the method is modified, edit and update the method on a 3.5 inch disk.

Step 1: Process 6 samples using the following procedure:

Step 2: Condition column with 10 mL of acetonitrile into SOLVENT WASTE

Step 3: Condition column with 5 mL of acetonitrile into SOLVENT WASTE

Step 4: Condition column with 5 mL of water into AQUEOUS WASTE

Step 5: Condition column with 5 mL of HOAc/water 0.1% into AQUEOUS WASTE

Step 6: Load 80 mL of sample onto column

Step 7: Rinse column with 5 mL of HOAc/Water 0.1% into AQUEOUS WASTE

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Step 8: Dry Column with gas for 0.5 minutes

Step 9: Wash syringe with 5 mL of HOAc/MeCN 0.1%

Step 10: Collect 5 mL fraction into sample tube using HOAc/MeCN 0.1%

Step 11: END

FLOW RATES	
Conditioning flow	15.0 mL/min
Load flow	10.0 mL/min
Rinse flow	20.0 mL/min
Elute flow	5.0 mL/min
Conditioning Air Push	15.0 mL/min
Rinse Air Push	20.0 mL/min
Elute Air Push	5.0 mL/min
SPE PARAMETERS	
Push Delay	5 sec
Air Factor	1.0
Autowash Volume	1.00 mL
WORKSTATION PARAMETERS	
Maximum Elution Volume	12.0 mL
Exhaust Fan on	Y Y=Yes N=No
Beeper on	Y Y=Yes N=No
NAME SOLVENTS	
Solvent 1	water
Solvent 2	methanol
Solvent 3	acetonitrile
Solvent 4	HOAc/Water 0.1%
Solvent 5	HOAc/MeCN 0.1%

HOAc/Water = acetic acid in water, 0.1% v/v
HOAc/MeCN = acetic acid in acetonitrile, 0.1% v/v
gas = compressed air or nitrogen

NEVER USE ACETONE ON THE ZYMARK AUTOTRACE SPE.

12.3.10 After the system is completed, the screen displays "program completed". Move the extracts aside and seal them with a teflon-lined screw cap. Remove the SPE columns and discard them, and record the final mass of the sample bottles and caps.

12.3.11 Run the 'Cleaning Sample Lines program' to clean sample lines, using emptied columns.

12.3.12 After the lines are cleaned, either continue with the next set of samples or turn off the Zymark - place the sample lines in a plastic bag.

12.3.13 Adjust the final volume to 5.0 mL with acetonitrile, and add 5 uL of concentrated acetic acid to stablize TETRYL. The extract concentration is 80 mL/5 mL.

12.3.14 Mix the contents well, and filter the acetonitrile extract through an Acrodisc LC25 filter assembly using a 10-cc disposable syringe into a new test tube. DO NOT READJUST THE VOLUME.

12.3.15 The acetonitrile extracts are stored in the freezer. A dilution of 1/10 is required for analysis to obtain a final concentration of 80mL/50mL.

12.3.15.1 For HPLC/UV, dilute 0.4 mL of the extract with 3.6 mL of distilled water in an 4-mL vial when ready for analysis.

12.3.15.2 For LC/TSP/MS, dilute 0.4 mL of the extract with 3.6 mL of 0.2 M ammonium acetate in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix the contents well. The extracts are stored in the freezer until ready for analysis by LC/TSP/MS.

12.4 LOW LEVEL salting-out method; for 8330 by HPLC/UV and 8330/SPEX by LC/TSP/MS.

This method is not applicable for nitroguanidine and picric acid - use DAI.

The efficiency of extraction depends on the size of the stirring bar, the speed of the magnetic stirrer, and the design of the volumetric flask as the extraction vessel.

It is imperative that the amount of water sample, the mass of the sodium chloride, and the amount of acetonitrile be ACCURATELY measured and used as specified in this method.

12.4.1 Label the sample tape with the appropriate suffix '8330-LOW-A' for 8330 by HPLC/UV or 8330/SPEX-LOW-A for 8330 and specialty explosives by LC/TSP/MS.

12.4.2 Measure 770 mL of water sample into a 1000-mL volumetric flask by weight.

12.4.2.1 For control, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.4.3 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.4.3.1 For 8330 by HPLC/UV, add 40 uL of the 50 ug/mL surrogate solution to the 770 mL water samples to yield 2.6 ppb.

12.4.3.2 For 8330/SPEX by LC/TSP/MS, add 80 uL of the 50 ug/mL standard of d_5-Nitrobenzene to the 770 mL water samples to yield 5.2 ppb.

12.4.4 If applicable fortify the DCS, LCS, and the MS/MSD.

12.4.4.1 For 8330 by HPLC/UV, add 77 uL of the 5.0 ug/mL 8330-Mix standard to the 770 mL water samples to yield 0.50 ppb.

12.4.4.2 For 8330/SPEX by LC/TSP/MS, add 77 uL of the 5.0 ug/mL 8330 mix and 77 uL of the 20/20/10 ug/mL NGl/PA/PETN mix to the 770 ml water samples to yield 0.50 ppb on the 8330 mix and 2.0/2.0/1.0 ppb on the SPEX mix.

12.4.5 Add 251.4 grams of sodium chloride (± 0.2 grams) to each water sample in the volumetric flasks, including the control and spikes. Carefully add the stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt dissolves.

12.4.6 Add 164 mL of acetonitrile (measured with a 250-mL graduated cylinder) while the solution is being stirred and stir for an additional 15 to 20 minutes at high speed. An efficient stirring is observed when the acetonitrile layer is vortexing in the water sample.

12.4.7 When the stirring is completed, turn off stirrer and allow the phases to separate for a minimum of 10 minutes.

12.4.8 Carefully transfer the upper acetonitrile phase (about 8 mL) to a 100-mL volumetric flask containing the 7/8 inch stirring bar with a pasteur pipet.

12.4.8.1 The amount of acetonitrile recovered should be approximately 6 to 10 mL. A larger volume could be indicative of inefficient vortexing of the extraction solvent into the water sample.

12.4.9 Extract the aqueous sample once more with another 8 to 10 mL of acetonitrile, stir at high speed for 15 to 20 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the 100-mL volumetric flask. The inclusion of a few drops of salt water at this point is not important.

12.4.10 Add 84-85 mL of salt water (325 g sodium chloride per 1000 mL of distilled water) to the extracts in the 100-mL volumetric flask and stir the contents on the magnetic stirrer for 15 to 20 minutes at high speed. Again, an efficient stirring is observed when the acetonitrile layer is vortexing in the salt water.

12.4.10.1 After stirring for 5 minutes, allow the phases to separate to determine if any acetonitrile is recoverable. Occassionally there is little or no acetonitrile layer visible but the sample is not lost. Add an extra 1 to 4 mL of acetonitrile, and stir the solution again.

12.4.11 When the stirring is completed, turn off stirrer and allow the phases to separate for a minimum of 10 minutes.

12.4.12 Carefully transfer the upper acetonitrile phase (about 3 to 4 mL) to an 8-mL test tube with a pasteur pipet (and minimize the amount of water being transferred with the acetonitrile).

12.4.13 Extract the salt water once more with another 1.0 mL of acetonitrile, stir at high speed for 15 to 20 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the test tube.

12.4.13.1 If the volume of acetonitrile from the first extraction in the 8-mL test tube is less than 4 mL, then use 1 mL of acetonitrile on the second extraction (not to exceed 5.0 mL final volume).

12.4.13.2 If the volume of acetontrile is over 4 mL, use less acetonitrile; do not exceed 5 mL.

12.4.13.3 If the volume of acetonitrile is over 5 mL, proceed with the second extraction with 1 mL acetonitrile, then transfer the combined acetonitrile in another 100-mL volumetric flask quantitatively with acetonitrile rinses, and proceed with steps 12.4.10 to 12.4.13 to reduce the volume.

12.4.14 Carefully adjust the volume to 5.0 mL with acetonitrile (NO nitrogen blowdown permitted), and add 5 uL of concentrated acetic acid to stablize TETRYL. Mix the contents well.

C4-98

12.4.15 Filter the extract through a 25-mm PTFE 0.45 um filter assembly using a 10-cc disposable syringe into another 8-mL test tube.

12.4.16 The acetonitrile extracts are stored in the freezer. A dilution of 1/4 is required for analysis to obtain a final concentration of 770mL/20mL.

12.4.16.1 For HPLC/UV, dilute 1.0 mL of the extract with 3.0 mL of distilled water in an 8-mL test tube when ready for analysis.

12.4.16.2 For LC/TSP/MS, dilute 1.0 mL of the extract with 3.0 mL of 0.2 M ammonium acetate in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix the contents well. The extracts are stored in the freezer until ready for analysis by LC/TSP/MS.

12.5 **LOW LEVEL SPE method - EXTRACTION ON THE ZYMARK AUTOTRACE WORKSTATION**

SPE column, 6-mL, 500 mg Porapak RDX Cartridges, Waters Catalog number WAT047220, or equivalent.

12.5.1 Label the sample tape with the appropriate suffix '8330-LOW-A' or '8330/SPEX-A'.

12.5.2 QC's setup (MB, DCS, LCS, and MS/MSD) for Zymark AutoTrace SPE

12.5.2.1 For MB, DCS, and LCS, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.5.2.2 If requested, control water supplied by the client may be used for MS/MSD. See section 12.5.4.1 if sample contains sediments or suspended particulates.

12.5.2.3 Measure 900 mL of water sample by weight into a 1-L amber glass bottle for QC's.

12.5.3 If applicable, fortify the DCS, LCS, and MS/MSD.

12.5.3.1 For 8330 by HPLC/UV, add 90 uL of the 5.0 ug/mL 8330 Mix standard to the 900 mL water samples t yield 0.5 ppb.

12.5.3.2 For 8330/SPEX by LC/TSP/MS, add 90 uL of the 5.0 ug/mL 8330 Mix standard and 90 uL of the 20/20/10 ug/mL NGl/PA/PETN Mix to the 900 mL water samples to yield 0.5 ppb on the 8330 and 2/2/1 ppb on NGl/PA/ and PETN.

12.5.4 Measure 900 ml of aqueous samples by weight into glass bottles in order to be able to add surrogate to the samples.

12.5.4.1 If the water sample contains sediments or suspended particles, the samples must be separated from its particulates by centrifuging or by decanting into a cleaned 1-L amber glass bottle.

12.5.5 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.5.5.1 For 8330 by HPLC/UV, add 45 uL of the 50 ug/mL surrogate solution to the 900 mL water samples to yield 2.5 ppb.

12.5.5.2 For 8330 by LC/TSP/MS, add 100 uL of the 50 ug/mL d_5-nitrobenzene standard to the 900 mL water samples to yield 5.6 ppb.

12.5.6 Weigh and record the initial mass of the sample bottles, including the QC's (with the screw caps on).

12.5.7 Samples ready for extraction by the Zymark Autotrace SPE.

12.5.8 The program for the Autotrace SPE workstation must be set up using the PC computer with the following parameters on a 3.5" disk.

12.5.9 Below is an overview of the program. Whenever the method is modified, edit and update the method on a 3.5 inch disk.

Step 1: Process 6 samples using the following procedure:

Step 2: Condition column with 10 mL of acetonitrile into SOLVENT WASTE

Step 3: Condition column with 5 mL of acetonitrile into SOLVENT WASTE

Step 4: Condition column with 5 mL of water into AQUEOUS WASTE

Step 5: Condition column with 5 mL of HOAc/water 0.1% into AQUEOUS WASTE

Step 6: Load 800 mL of sample onto column

Step 7: Rinse column with 5 mL of HOAc/Water 0.1% into AQUEOUS WASTE

Step 8: Dry Column with gas for 0.5 minutes

Step 9: Wash syringe with 5 mL of HOAc/MeCN 0.1%

Step 10: Collect 5 mL fraction into sample tube using HOAc/MeCN 0.1%

Step 11: END

FLOW RATES

Conditioning flow	15.0 mL/min
Load flow	10.0 mL/min
Rinse flow	20.0 mL/min
Elute flow	5.0 mL/min
Conditioning Air Push	15.0 mL/min
Rinse Air Push	20.0 mL/min
Elute Air Push	5.0 mL/min

SPE PARAMETERS		
Push Delay	5	sec
Air Factor	1.0	
Autowash Volume	1.00 mL	
WORKSTATION PARAMETERS		
Maximum Elution Volume	12.0 mL	
Exhaust Fan on	Y	Y=Yes N=No
Beeper on	Y	Y=Yes N=No
NAME SOLVENTS		
Solvent 1	water	
Solvent 2	methanol	
Solvent 3	acetonitrile	
Solvent 4	HOAc/Water 0.1%	
Solvent 5	HOAc/MeCN 0.1%	

HOAc/Water = acetic acid in water, 0.1% v/v
HOAc/MeCN = acetic acid in acetonitrile, 0.1% v/v
gas = compressed air or nitrogen.

NEVER USE ACETONE ON THE ZYMARK AUTOTRACE SPE.

12.5.10 Zymark Autotrace process time for six samples is approximately 124 minutes.

12.5.11 Before starting for the day, solvent lines should be purged by loading the solvent purging program. Check the solvents and replace the solvents if necessary.

12.5.12 The sample lines should have been in a plastic bag, indicating the lines have been cleaned with methanol followed by distilled water from the Cleaning Sample Lines program.

12.5.13 Set the SPE columns in place and depress the plunger. Once in place the green light should be on.

12.5.14 Set the receiving vessels (test tubes) in place. Be sure the first sample is on your left and the sixth sample on your right.

12.5.15 Check the solvents - replace if neccessary, and set the samples in its place.

12.5.16 Check the waste containers (Aqueous and Organic) and replace if necessary. Do NOT allow waste containers to overflow.

12.5.17 Load the program '8330-A' in the disk drive of the Zymark Autotrace, and press Load.

12.5.18 When the disk has been loaded, the Zymark Autotrace is ready to start. Follow instructions displayed on the screen "Press CONT to start" if ready to start.

12.5.19 After the system is completed, the screen displays "program completed". Move the extracts aside and seal them with teflon-lined screw caps. Remove the SPE columns and discard them, and record the final mass of the sample bottles and caps.

12.5.20 Run the 'Cleaning Sample Lines program' to clean sample lines, using emptied columns.

12.5.21 After the lines are cleaned, either continue with next set of samples or turn off the Zymark - place the sample lines in a plastic bag.

12.5.22 Adjust the final volume to 5.0 mL with acetonitrile, and add 5 uL of concentrated acetic acid to stablize TETRYL. Mix the contents well. The extract concentration is 800mL/5mL.

12.5.23 Filter the acetonitrile extract through an Acrodisc LC25 filter assembly using a 10-cc disposable syringe into a new test tube. DO NOT READJUST THE VOLUME.

12.5.24 The acetonitrile extracts are stored in the freezer. A dilution of 1/4 is required for analysis to obtain a final concentration of 800mL/20mL.

12.5.24.1 For HPLC/UV, dilute 1.0 mL of the extract with 3.0 mL of distilled water in a 4-mL vial when ready for analysis.

12.5.24.2 For LC/TSP/MS, dilute 1.0 mL of the extract with 3.0 mL of 0.2 M ammonium acetate in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix the contents well. The extracts are stored in the freezer until ready for analysis by LC/TSP/MS.

12.6 Soil/Sediment Samples.

NOTE: Do NOT grind samples containing high concentrations of explosives. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

12.6.1 Subsample approximately 20 to 50 grams of soil into a 16-oz wide mouth jar for drying.

12.6.2 If the samples do not appear homogeneous and not suspicious, dry the samples in a cool, ventilated area and away from direct light. DO NOT HEAT the samples.

12.6.2.1 If samples do appear suspicious, stop work and consult with the project manager and client regarding to possible high levels of explosives.

12.6.3 After the samples are dried, grind thoroughly in mortar with pestle (both items previously rinsed with acetonitrile and dried).

12.6.4 Sift the sample through a sieve size 30 mesh. If samples are not to be extracted on the same day, store the sifted soil in the freezer and protect from light.

12.6.5 Label the sample tape with "8330-S" for 8330 by HPLC/UV, "NGU-S" for Nitroguanidine, "SPEX-S" for specialty explosives (Nitroglycerine, PETN, and Picric Acid), and 8330/SPEX-S for 8330 mix and specialty explosives by LC/TSP/MS.

12.6.5.1 Label the sample tape with 'PA-S' for picric acid as this is a separate extraction.

12.6.6 When the extractions are to be performed, turn on the recirculator cooler to cool the sonication bath to operating temperature (5 to 25 ^{o}C). The sonicator bath generates heat when the sonication bath is on.

12.6.7 Weigh 2.0 grams of homogeneous soil into a 40-mL vial.

12.6.7.1 For control, use Ottawa sand. If a larger sample size is desired, then the ratio of sample to solvent must be consistent to 1 gram/5 mL.

12.6.7.2 For picric acid, weigh out a separate set.

12.6.8 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.6.8.1 No surrogate is used for the nitroguanidine analysis.

12.6.8.2 For 8330 by HPLC/UV, add 80 uL of the 50 ug/mL surrogate solution to each 2.0 gram soil sample to yield 2.0 ppm.

12.6.8.3 For 8330/SPEX and SPEX by LC/TSP/MS, add 200 uL of the 50 ug/mL d_5-Nitrobenzene standard to each 2.0 gram soil sample to yield 5.0 ppm.

12.6.8.4 For picric acid, add 50 uL of the 50 ug/mL d_5-Nitrobenzene standard to each 2.0 gram soil sample to yield 1.25 ppm.

12.6.9 If applicable, fortify the DCS/LCS, and MS/MSD.

12.6.9.1 For nitroguanidine, fortify at 1.0 ppm with Nitroguanidine by adding 40 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.2 For 8330-HPLC/UV, fortify at 1.0 ppm with 8330 mix by adding 40 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.3 For 8330 by LC/TSP/MS, fortify at 2.0 ppm with 8330 mix by adding 80 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.4 For SPEX-LC/TSP/MS, fortify at 5/5/2.5 ppm of NGl/PA/PETN with SPEX Mix by adding 0.50 mL of the 20/20/10 mix to the 2 gram samples.

12.6.9.5 For PA in soils using the method for picric acid, fortify at 1.25 ppm with SPEX Mix by adding 125 uL to the 2 gram samples.

12.6.10 Adjust the final volume to 10.0 mL with 0.1% (v/v) acetic acid in acetonitrile (except for 'PA-S' see below). Cap, and shake briefly to mix the contents well.

12.6.10.1 To the separate set of PA-S (picric acid in soil) adjust the final volume to 10.0 mL with 0.1% hydrochloric acid in 10% acetonitrile in water, cap, and shake briefly to mix the contents well.

12.6.11 Place the samples in the ultrasonic bath, and sonicate for 18 hours, keeping the bath at 35 °C or cooler.

12.6.11.1 Record the initial time, date, and temperature of the water bath on the benchsheet. Cover the sonication bath with aluminum foil or a cover to protect samples from light.

12.6.11.2 After sonication, record the final time, date, and temperature of the water bath.

12.6.12 Allow the samples to settle for 30 minutes or centrifuge the samples at approximately 1000 rpm for about 5 minutes.

12.6.13 Decant the extracts (minimally 4 mLs) into 8-mL test tubes. The extracts may be worked up immediately or stored in the freezer until ready for further prep.

12.6.14 Filter the required amount needed for analysis by transferring into a 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 8-mL screwcap test tubes.

12.6.15 For 8330-HPLC/UV, dilute 1.0 mL of the filtered extract in an 8-mL screwcap test tube with 3.0 mL of the 1.3 g/L calcium chloride solution. Shake briefly to mix the contents.

12.6.15.1 Filter the extracts by transferring into a 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 4-mL screwcap vials.

12.6.15.2 Store the 8330-HPLC/UV extracts in the freezer and protect from light until analysis. The final sample concentration is 0.20 g/4.0 mL.

12.6.16 For nitroguanidine (NGu-S), dilute 2.0 mL of the filtered extract into an 8-mL test tube with 0.5 mL of distilled water to obtain a 80:20 acetonitrile:water composition. Mix the contents well.

12.6.16.1 Filter the extracts by transferring into 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into clean 4-mL screwcap vials.

12.6.16.2 Store the NGu-S extracts in the freezer and protect from light until analysis. The final concentration is 0.4g/2.5 mL.

12.6.17 For SPEX-LC/TSP/MS and 8330/SPEX-LC/TSP/MS, dilute 1.0 mL of the filtered extracts in an 8-mL test tube with 3.0 mL of distilled water (do NOT add calcium chloride solution!).

12.6.17.1 Add 20 uL of the internal standard mix d_3-2,6-DNT and d_4-1,3-DNB, and mix the contents well.

12.6.17.2 Filter the extracts with 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters into clean 4-mL screwcap vials. The final extract concentration is 0.2g/4.0mL.

12.6.17.3 Store the extracts in the freezer and protect from light until ready for analysis by LC/TSP/MS.

12.6.18 For PA-S, the extracts may be filtered or decanted into an 8-mL test tube, and the extracts may be worked up immediately or stored in the freezer until ready for further prep.

Since the extracting solvent is 0.1% hydrochloric acid in 10% acetonitrile in water, the organic content of the extract is less than 20% in water, therefore no dilution with water is required. The final extract concentration is 2.0g/10mL.

12.6.18.1 If the extracts has not been filtered, then filter the required amount needed for analysis with 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters into clean 8-mL screwcap test tubes.

12.6.19 Transfer 2.0 mL of the filtered extracts into 4-mL vials, and add 10 uL of the internal standard mix d_3-2,6-DNT and d_4-1,3-DNB to yield 50 ng/mL in the final extract. Mix the contents well, and the final extract concentration is 0.4g/2.0mL.

12.6.20 Store the extracts in the freezer and protect from light until analysis by LC/TSP/MS.

12.7 **INSTRUMENTATION for nitroguanidine by HPLC/UV**

The conditions listed below are the recommended analytical conditions for nitroguanidine by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.7.1 Primary Column: Phenomenex Zorbax C_8, 250 x 4.6 mm, 5 um, or equivalent.

12.7.2 Column Temperature: 30 °C

12.7.3 Mobile Phase (Reservoir)
A = Water
B = Acetonitrile
C = Methanol

Gradient Program:

Time	A	B	C
0.0	100	0	0
8.0	100	0	0
9.0	0	50	50
15.0	0	50	50

12.7.4 Flow rate: 1.0 mL/min.

12.7.5 Injection volume: 25 uL

12.7.6 UV Detection at 250 nm.

12.7.7 Approximate retention times on C_8 column:

Nitroguanidine 4.3 minutes

12.7.8 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response.

If nitroguanidine is not retained or the retention time is close to the void volume on the C8 or C18 column, replace with another column.

12.8 INSTRUMENTATION for 8330 by HPLC/UV

The conditions listed below are the recommended analytical conditions for 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.8.1 Primary Column: Phenomenex Hypersil C_8, 150 x 4.6 mm, 5 um, or equivalent.

12.8.2 Column Temperature: 20 °C (Column kept cooled to a constant tempeature with a recirculated cooler).

12.8.3 Mobile Phase (Reservoir)

A = Water
B = 98:2 (v/v) methanol/tetrahydrofuran
C = 98:2 (v/v) methanol/acetonitrile

Gradient program:

Time	A	B	C
0.0	75	25	0
2.0	75	35	0
22.0	35	0	65
24.0	0	0	100

12.8.4 Flow rate: 1.5 mL/min.

12.8.5 Injection volume: 200 uL on UV-9050
250 uL on UV-200

12.8.6 UV Detection at 250 nm.

12.8.7 Elution order and approximate retention times on C_8 column:

HMX	3.90 minutes	
TNB	6.59	
RDX	7.16	
2,4-DNFB	8.58	(Surrogate)
DNB	9.43	
NB	10.95	
TNT	12.03	
TETRYL	13.09	
2,4-DNT	14.22	
2,6-DNT	14.92	
2-AM-4,6-DNT	15.47	
4-AM-2,6-DNT	15.75	
2-NT	16.24	
4-NT	16.52	
3-NT	17.09	

12.8.8 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response. The use of guard column causes poor resolution between RDX and TNB; therefore, guard columns are not used.

Co-elution of RDX and TNB and of 2-AM and 4-AM on the primary column depends on the type and condition of the column and the solvent composition of THF in methanol.

Loss of resolution between TNB and RDX may be caused by column deterioration.

If the solvent composition of THF in methanol is changed, RDX and TNB elution order may be reversed or co-elution may occur.

12.9 **INSTRUMENTATION for Confirmation of 8330 by HPLC/UV**

The conditions listed below are the recommended analytical conditions for confirmation of 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.9.1 Confirmation Column: Zorbax Cyano CN, 250 x 4.6 mm, 5 um, or equivalent.

12.9.2 Column Temperature: 20 °C (Column kept cooled to a constant temperature with a recirculated cooler)

12.9.3 Mobile Phase (Reservoir) and Gradient Program

OPTION 1

A = Water
B = 98:2 (v/v) methanol/THF
C = 98:2 (v/v) methanol/MeCN

Time	A	B	C
0.0	60	0	40
17.0	40	60	0
19.0	0	100	0
35.0	0	100	0

OPTION 2

A = water
B = acetonitrile
C = methanol

Time	A	B	C
0.0	80	8	12
20.0	60	28	12
35.0	0	100	0

12.9.4 Flow rate: 1.0 mL/min.

12.9.5 Injection volume: 500 uL on UV-9050
250 uL on UV-200

12.9.6 UV Detection at 250 nm.

12.9.7 Elution order and approximate retention times on CN column:

OPTION 1			OPTION 2		
NB	12.20		NB	18.52	
DNB	13.16		DNB	20.07	
TNB	13.63		2,4-DNFB	20.94	Surrogate
2-NT & 4-NT	15.19		TNB	21.57	
3-NT	15.59		2-NT & 4-NT	23.82	
1C3NB	15.62	Surrogate	3-NT	24.25	
2,6-DNT	16.23		RDX	24.71	
2,4-DNT	16.68		2,4-DNT	25.78	
TNT	17.96		2,6-DNT	26.37	
4-AM-2,6-DNT	18.19		2-AM-4,6-DNT	27.29	
2-AM-4,6-DNT	18.79		4-AM-2,6-DNT	27.50	
RDX	20.42		TNT	28.73	
TETRYL	23.14		HMX	29.48	
HMX	24.52		TETRYL	31.00	

3-NT and 1C3NB co-elute on option 1.
1C3NB and RDX co-elute on option 2.
2-NT and 4-NT co-elute on both options.

12.10 **INSTRUMENTATION for 8330 by LC/TSP/MS**

The conditions listed below are the recommended analytical conditions for the analysis of specialty explosives and for the analysis or confirmation of 8330 compounds. If changes to the LC/TSP/MS conditions are necessary, they will be noted on the chromatogram.

12.10.1 Primary Column: Novapak C-8, 3.9 X 150 mm, 5 um, or equivalent.

12.10.2 Temperature: Ambient

12.10.3 Mobile Phase (Reservoir)

A = 0.20 M Ammonium acetate (aqueous) with 0.10% acetic acid
B = N/A
C = n-Propanol

Gradient program:

Time	Flow	A	C	Grad
init	1.00	95	5	--
4.0	1.00	85	15	6
7.0	1.00	70	30	6
7.1	0.80	70	30	6
14.0	0.80	50	50	6
15.0	0.80	95	5	11
19.0	1.00	95	5	11

(flow rate in ml/min)

The post column pump flow rate of solution A is set at 0.40 mL/min throughout the run.

12.10.4 Injection volume: 150 uL

12.10.5 Thermospray (TSP) conditions

The following thermospray conditions must be optimized whenever a new thermospray vaporizer is installed in order to provide good performance.

Vaporizer Temperature:	80 to 120 °C
Source Temperature:	280 to 350 °C
Repeller Voltage:	0 to 50 Volts
Discharge Voltage:	1200 Volts
Filament Current:	0 to 400 uA
Electron Energy:	0 to 500 eV

(Note: Negative ion analysis will usually require discharge voltage of 800 to 1000 V, filament current of 400 uA, and electron energy of 500 eV).

12.10.6 Mass Spectrometer (MS) scanning conditions

Conditions below describe analysis in negative-ion selective-ion-monitoring (SIM) mode. Some compounds (e.g. HMX and RDX) also respond in positive-ion mode. These compounds may be analyzed using positive-ion selective-reaction-monitoring (SRM) for confirmation purposes or when interfering compounds are present in negative-ion SIM analysis. Ions used in positive-ion SRM analysis will differ from those listed below and must be established by analysis of standard reference materials.

Compound (in approximate order of elution)	Ion	Retention Time (approximated)
HMX	355	3:39
Trinitrobenzene	213	5:53
RDX	281	6:12
1,3-Dinitrobenzene	168	8:54
d_4-1,3-Dinitrobenzene	172	8:48 (Int. Std)
Picric Acid	228	8:50
Trinitrotoluene	227	9:52
TETRYL	241	10:12
d_5-Nitrobenzene	128	10:49 (Surrogate)
Nitrobenzene	123	10:57
Nitroglycerine	241	11:06
2,4-Dinitrotoluene	182	11:48
2-Amino-4,6-dinitrotoluene	197	11:56
d_3-2,6-Dinitrotoluene	185	12:12 (Int. Std)
4-Amino-2,6-dinitrotoluene	197	12:12
2,6-Dinitrotoluene	182	12:14
2- & 4-Nitrotoluene	137	13:08
PETN	330	13:14
3-Nitrotoluene	137	13:23

2-NT and 4-NT co-elute by this method.

12.10.7 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response.

13. DATA ANALYSIS AND CALCULATIONS

13.1 Results are reported in ug/g or mg/Kg (ppm) for soil, unless otherwise instructed.

13.2 Results are reported as dry weight since soil samples are air dried, ground with motar and pestle, and sieved, unless specified by the client.

13.3 The concentration of each analyte and surrogate in a sample is calculated by using the appropriate formula:

Without using internal standard correction for HPLC/UV;

$$\text{Amount Found (ppb)} = \frac{CF_a \cdot A_x \cdot V_x}{W_s}$$

where CF_a = Average Calibration Factor in ng/mL.
A_x = Sample Total Area Counts.
V_x = Sample Extract Final volume in milliliters.
W_s = Initial Sample weight in grams.

Using internal standard correction for LC/TSP/MS;

$$\text{Amount Found (ppb)} = \frac{CF_a \cdot A_x \cdot V_x}{W_s} \times \frac{C_{is}}{A_{is}}$$

where C_{is} = Internal Standard Concentration in ng/mL.
A_{is} = Internal Standard Total Area Counts.

The results of soils in ppb (ng/g) are converted to ppm (ug/g) by dividing the result in ng/g by 1000;

$$\text{ug/g} = \frac{X \text{ ng/g}}{1000 \text{ ng/ug}}$$

14. METHOD PERFORMANCE

14.1 The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

15. POLLUTION PREVENTION

NONE

16. WASTE MANAGEMENT

16.1 Flammable Waste

16.1.1 Acetone, acetonitrile, hexane, and methanol waste generated from solvent rinsing of glassware is collected as "Flammable Waste" in 1-gallon plastic bottles.

16.1.2 The unused portion of the acetonitrile extracts from soil extraction may be decanted from the soils and discarded as flammable waste. For the soil portion see "solid waste" below.

16.1.3 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "Flammable Waste" in the H-3 room.

16.1.4 Methylene chloride should be collected separately, see "DCM Waste" below. Any organic waste that is less than 50% methylene chloride is flammable waste.

16.2 Methylene Chloride (DCM) Waste

16.2.1 Methylene chloride, or dichloromethane (DCM), generated from solvent rinsing of glassware is collected separately from the Flammable Waste into "DCM Waste" in 1-gallon plastic bottles.

16.2.2 Any organic waste that is in excess of 50% methylene chloride is considered DCM waste.

16.2.3 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "DCM Waste" in the H-3 room.

16.3 Liquid-liquid extraction (LLE) Waste

16.3.1 Aqueous samples from high level and low level extraction (liquid-liquid extraction) after completion have been saturated with organic solvent (such as acetonitrile) is collected into 5-gallon carboy containers, or if the pH of the water samples were neutral (pH between 6 to 9) they can be collected directly into 55-gallon drum labelled "LLE Waste" in the H-3 room.

16.3.2 Aqueous samples collected in 5-gallon carboy containers are transferred to 55-gallon drum labelled "LLE Waste" in the H-3 room after the pH is check and ajusted if necessary to between 6 to 9.

16.4 High Performance Liquid Chromatography (HPLC) Effluent

16.4.1 Waste generated from the HPLC is collected into 1-L plastic bottles labelled "HPLC Waste", it consists primarily of methanol, acetonitrile, water, and ammonium acetate.

16.4.2 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "HPLC/ETHER Waste" in the H-3 room.

16.5 Solid Waste

16.5.1 Residual soil are allowed to air-dry in the hood or in well ventilated area. After the soils are dried, they are discarded into the "Dry Hazard" trash.

16.5.2 Dry Hazard trash consists of glass, gloves, papers, and plastics that were in contact with samples. When the trash is full, it is double bagged, secured, and disposed in the 55-gallon drum labelled "Dry Hazards" in the waste room where they are compacted in the 55-gallon drums.

16.5.3 Empty glass solvent bottles are disposed of in the waste room after they have been air dried. They are placed in the 55-gallon drum where they are compacted and crushed.

17. REFERENCES

17.1 SW-846 Draft Method 8330, "Nitroaromatics and Nitramines by High Pressure Liquid Chromatography (HPLC)", Revision 0, November 1992, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

17.2 Thomas F. Jenkins, et. al., "Comparison of Cartridge and Membrane Solid-Phase Extraction with Salting-Out Solvent Extraction for Preconcentration of Nitroaromatic and Nitramine Explosives from Water", Special Report 92-95, December 1992, US Army Corps of Engineers, Cold Regions Research & Engineering Laboratory, Hanover, New Hampshire.

17.3 "Quanterra Quality Control Program", QA-003, Quanterra Environmental Services, Revision No. 0, April 4, 1995.

17.4 "Laboratory Documentation Practices", LP-CAL-0001, Quanterra Environmental Services, Revision 3.0, December 5, 1991.

17.5 "Calibration and Calibration Check of Balances", SAC-QA-0041, Quanterra Environmental Services, Revision 1.1, December 30, 1994.

18. MODIFICATIONS TO SOP

18.1 This method deviates from EPA method 8330, November 1992 revision on the following:

Acetonitrile is our preferred solvent instead of methanol in the preparation of standards.

The final solvent for extracts for HPLC/UV analysis should be no more than 25% acetonitrile in water. Higher percentages of acetonitrile causes poor chromatography.

Sodium chloride is used in the salting out method of aqueous samples instead of sodium sulfate.

The primary column used in this method is C-8 reverse phase instead of C-18, and the column length is 15 cm instead of 25 cm.

A gradient program is run using Water/Methanol with THF and Acetonitrile as modifiers instead of isocratic at 50/50 Methanol/Water.

The initial calibration is performed by injecting five concentration levels singly instead of triplicate injections of five levels randomly.

The amount of calcium chloride solution (5g/L aqueous) added to the soil extracts is 2:8 and is less than the original method of 1:1. Calcium chloride solution is a flocculating agent that aids in the filtration of soil extracts. Higher concentrations may cause clogging of the frits and over-pressurizing the HPLC.

The UV detector is set at 250 nm instead of 254 nm.

The injection volume is 200 uL instead of 100 uL.

18.2 Changes made on the original revision to revision 1.0.

Improved the stability of TETRYL in soil by the addition of acetic acid at 0.1% by volume in acetonitrile as the extraction solvent.

Included the component Nitroguanidine by HPLC/UV.

Included the components Nitroglycerine, PETN, and Picric Acid by LC/TSP/MS.

8330 components can also be analyzed by LC/TSP/MS.

Nitroglycerine, PETN, and Picric acid can be extracted using 0.1% hydrochloric acid in acetonitrile instead of acetic acid in acetonitrile; however, nitroglycerine and PETN can be extracted using acetic acid along with 8330 components.

18.3 Changes made on revision 1.0 to revision 2.0.

Added sections to include extraction of 8330 components at high levels by the low level extraction.

Included the extraction of Nitroguanidine in water and in soil.

Included the extraction of Specialty Explosives (Nitroglycerine, Picric Acid, and PETN) in water and in soil.

Included the LC/TSP/MS conditions for Specialty Explosives.

Analysis may be performed by LC/TSP/MS.

Confirmation on positives measured by HPLC/UV may be performed by LC/TSP/MS.

18.4 Changes made on revision 2.0 to revision 3.0

Component 1-Chloro-3-nitrobenzene is added as a surrogate for 8330 by HPLC/UV analysis.

Component d_5-Nitrobenzene is added as a surrogate for 8330 by LC/TSP/MS and component d_4-1,3-Dinitrobenzene is added as another internal standard with d_3-2,6-Dinitrotoluene for LC/TSP/MS.

Combining method 8330 and specialty explosives as a single methodology by LC/TSP/MS (8330/SPEX).

Included flow diagrams.

18.5 Changes made on revision 3.0 to revision 4.0.

Updated to Quanterra format.

Correction on the naming of TETRYL as Methyl-2,4,6-trinitrophenylnitramine, not Methyl-2,4,6-trinitrophenylamine.

Analytical curve and midpoint standards for 8330 by HPLC/UV are prepared in 25:75 0.1% acetic acid in acetonitrile:HPLC grade water, and is valid for up to 45 days when properly stored in the refrigerator.

Addition of spiking soils for 8330-LCMS with 8330 mix at 2.0 ppm.

Changed final solvent composition of SPEX-A and 8330/SPEX-A to 20% acetonitrile in water (injection of samples greater than 20% acetonitrile in water adversely affects the chromatography).

Correction and clarity on the final prep of 8330/SPEX-LCMS-A.

Soil extracts are decanted into 8-cc test tubes for storage after extraction.

Addition of Method 8330 by SPE as Low Level and High Level in place of salting-out method.

Injection volume for cyano confirmation on UV 9050 to 250 uL and on UV 100 to 370 uL.

Injection volume for analytical column on UV 100 to 250 uL.

Separate extraction method for picric acid with 0.1% hydrochloric acid in 10% acetonitrile in water.

18.6 Changes made on revision 4.0 to revision 5.0.

SOP number changed from SAC-GC-0124 to SAC-LC-0001.

Other references related to quality added to References section.

Tetrahydrofuran added to list of flammable solvents.

Added TNT/RDX Soil Extrtaction Pac to test suspected soils before grinding.

New surrogate added, 2,4-dinitrofluorobenzene (2,4-DNFB) for HPLC/UV.

Changed the concentration range of the new surrogate for the initial standard curve on HPLC/UV.

Changed the standard range up to 1000 ng/mL for 8330 and eliminated level 4 standard on HPLC/UV.

Modify aqueous samples for the stability of TETRYL by adding 5 uL of concentrated acetic acid to the final 5.0 mL acetonitrile extracts.

Changed the concentration range of d5-Nitrobenzene for the initial standard curve on LC/TSP/MS.

Adjusted the spike level of d5-Nitrobenzene on aqueous samples to achieve 200 ng/mL in the final extracts.

Correction on UV-100 as UV-200.

Correct injection volume for cyano confirmation on UV 200 at 250 uL, not 370 uL.

Triplicate injection is no longer required at the begining of the batch but a single injection and it is to be less than or equal to 15% of the expected value from the initial calibration curve.

All continuing calibration standards in the batch are to be less than or equal to 15% of the initial calibration curve and not from the triplicate standard injections.

Soils are dried until no visible appearance of moisture present. Successful grinding and sifting verifies that soils are dried.

Updated instrument conditions for 8330 by HPLC/UV and for 8330 confirmation by HPLC/UV.

Added "LLE Waste" under Waste Management section to handle aqueous waste saturated with organic solvent (acetonitrile used in the high level and low level salting out methods).

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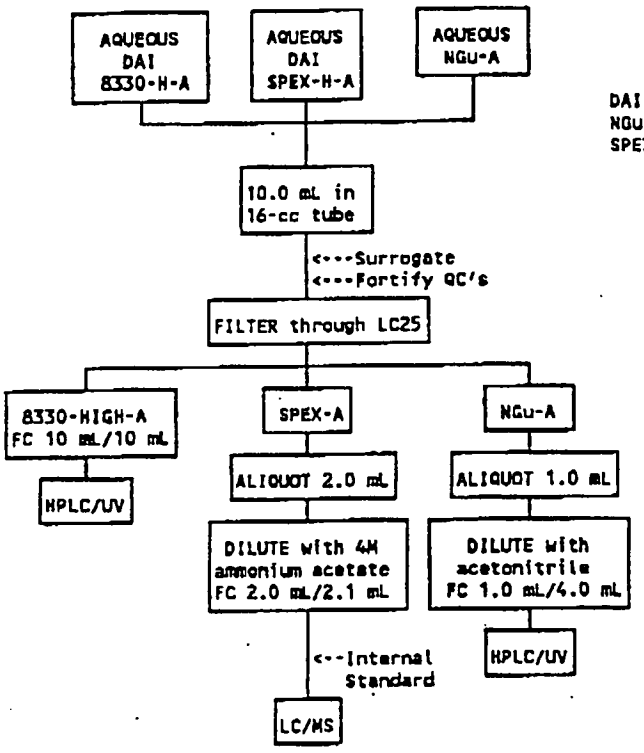
19. FLOW DIAGRAM

19.1 Aqueous by DAI for high levels.



DAI = Direct Aqueous Injection
NGu = Nitroguanidine
SPEX = Specialty Explosives (Nitroglycerine, Picric Acid, and PETN)

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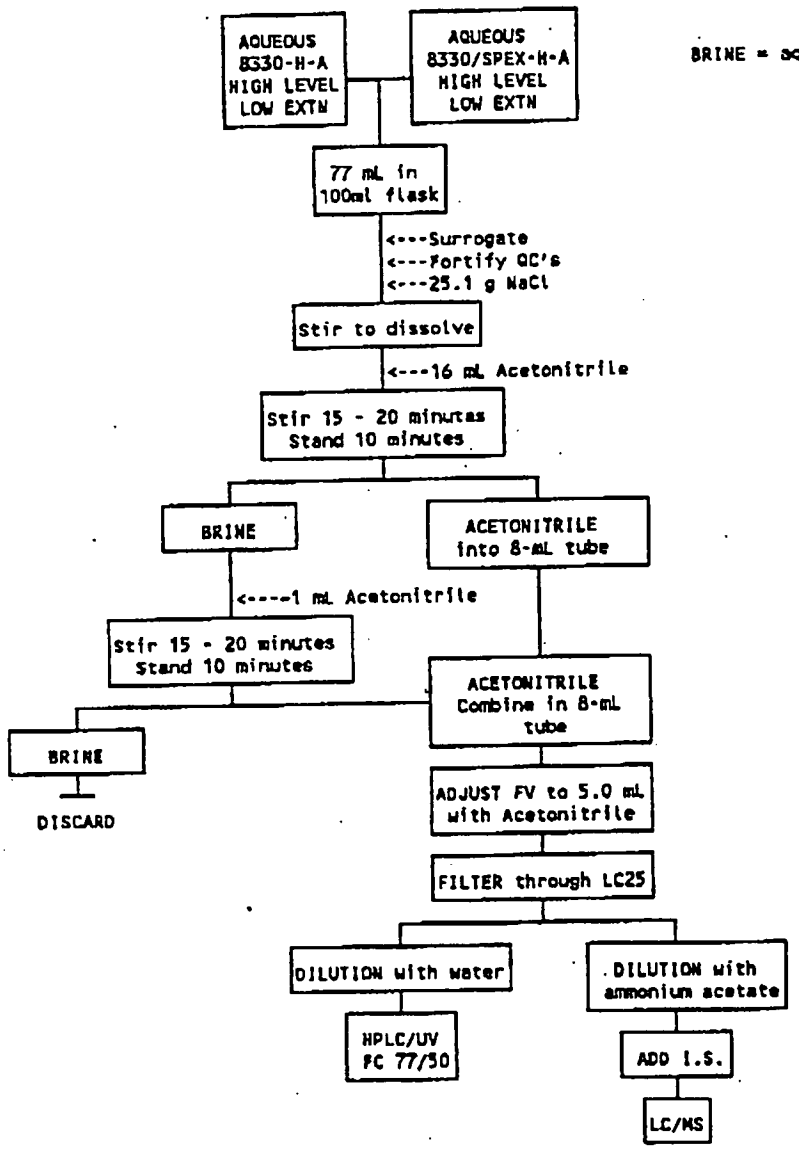
Revision No. 5.0
Date January 3, 1996
Page 59 of 63

19.2 Aqueous by Salting Out for high levels.



C4-125

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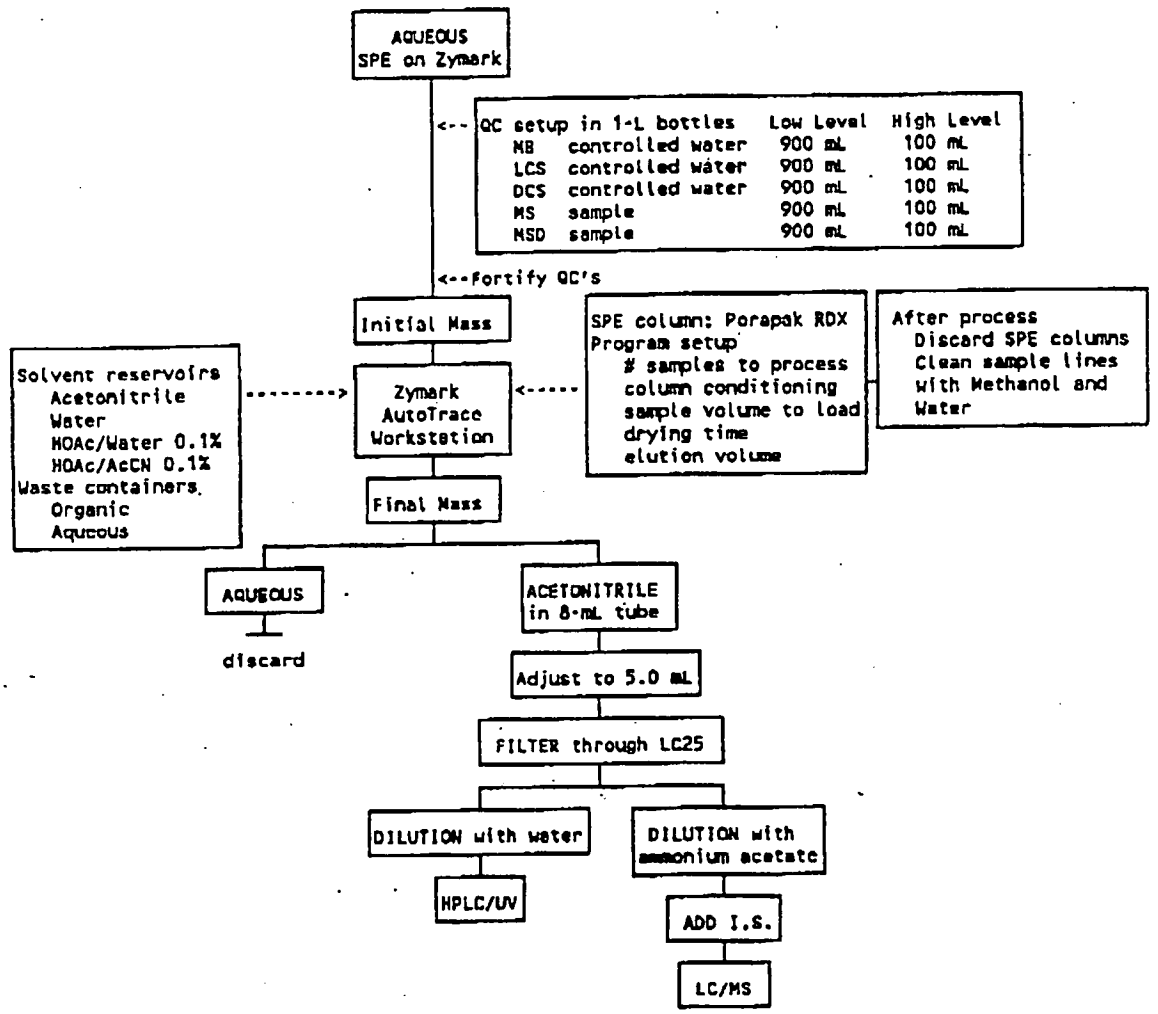
Revision No. 5.0
Date January 3, 1996
Page 60 of 63

19.3 Aqueous by SPE using Zymark Autotrace Workstation for high levels or low levels.



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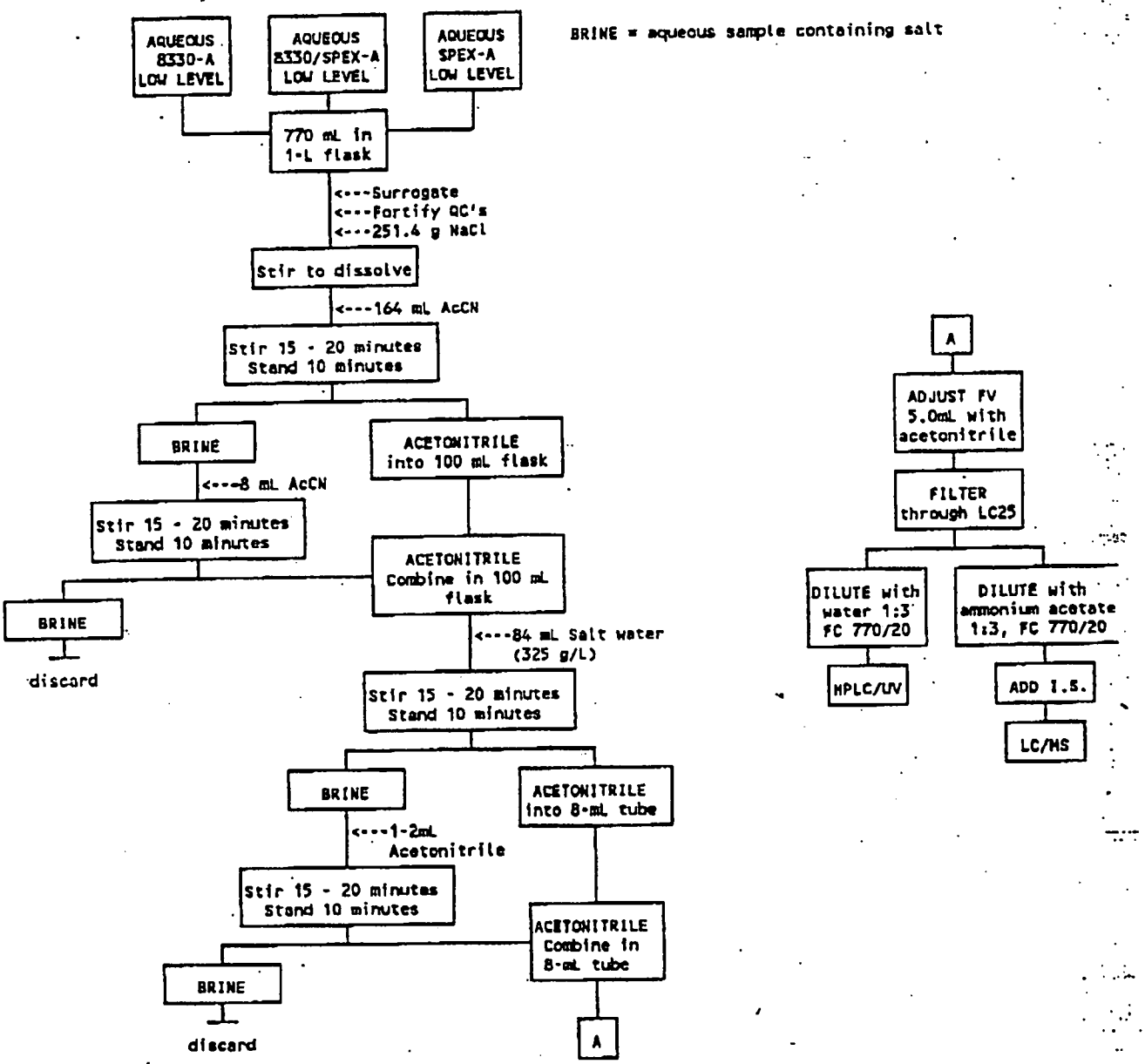
SOP No. SAC-LC-0001
Revision No. 5.0
Date January 3, 1996
Page 61 of 63

19.4 Aqueous by Salting Out for low levels.



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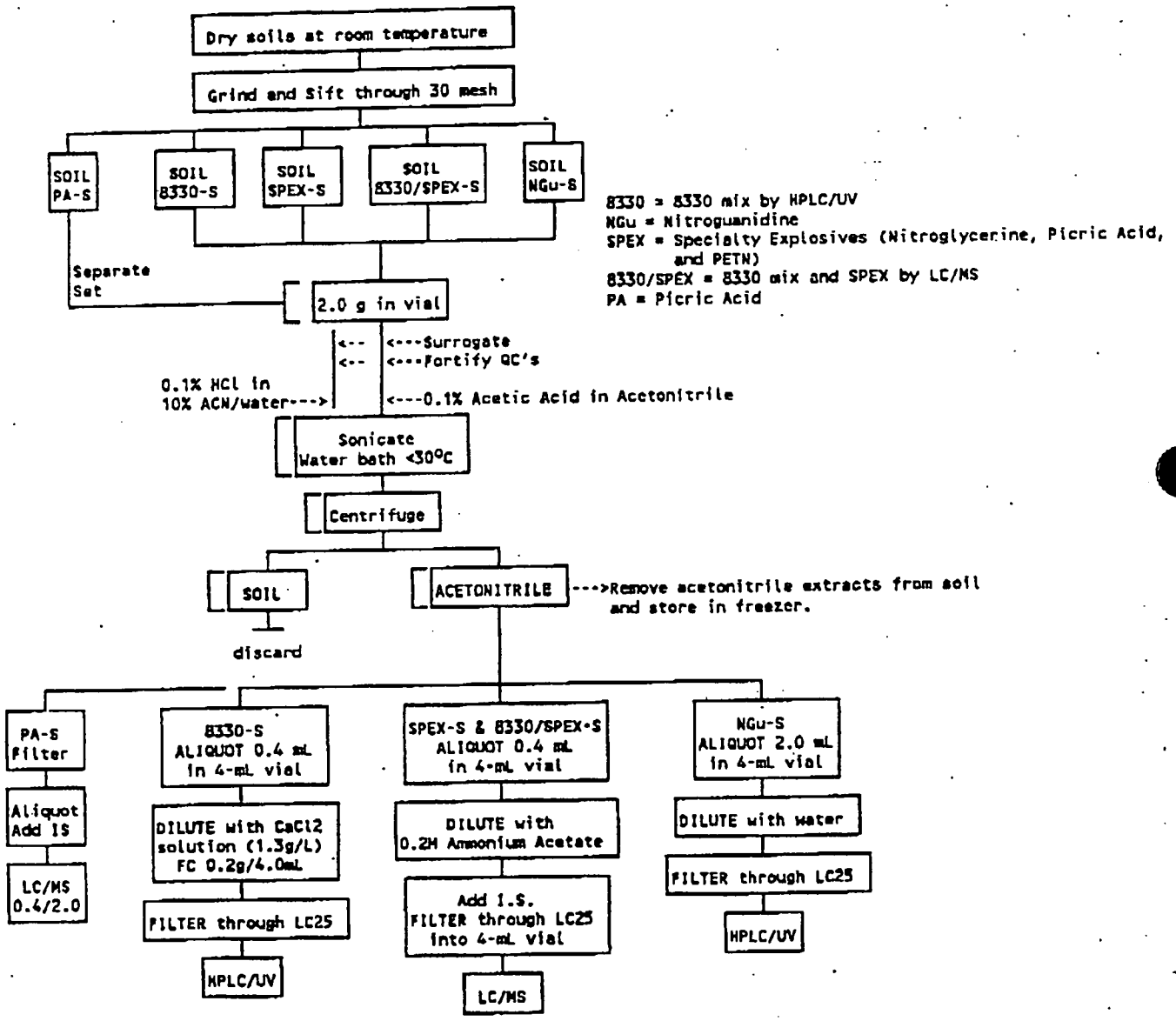
Revision No. 5.0
Date January 3, 1996
Page 62 of 63

19.5 Soil for 8330 and specialty explosives.



C4-128

20. APPENDIX

A copy of form - STAMDARD SOLUTION MADE FROM NEAT.

A copy of form - STANDARD SOLUTION MADE FROM SOLUTION(S).

A copy of form - MASTER SHEET

A copy of form - HPLC INSTRUMENT LOG

Quanterra / W. Sacramento

CHROMATOGRAPHY RUN LOG

Method: 8330_HY File: [MAY_SV] 4LC Inst: hplc #4 [109]

Calib: 4HY0513I 8330 Curve; 2.5 to 1000 ng/mL Hypersil C8 on 5/13/96

HPLC-UV #4 Phenomenex Hypersil C8 SN114198 150x4.6mm 5um, 8330 Analys

#	SAMPLE NAME	MULT	DIVS	DATE	TIME	LVL
1	Water Blank	1.00 /	1.000	20-MAY-1996	13:35	
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						
39						
40						

COLUMN	A -	B -

Analyst: _____ Date: _____ Page: ____

Q/WS Elog 1.1 Printed: 05/20/96 15:14:59

C4-130

MASTER SHEET

Holding time Due: _____ PROJECT Due: _____

PROJECT NO. : _____ Page ___ of ___

INITIATED BY: _____ DATE: _____

METHOD NO. : _____

SAMPLE MATRIX: _____

COMPOUND(S) : _____

COMPLETED BY: _____ DATE: _____

REVIEWED BY : _____ DATE: _____

APPROVED BY : _____ DATE: _____

STORAGE								

Comments: _____

CAL ID/SAMPLE DATA	SAMPLE SIZE	FORTIFICATION - STANDARD DATA PPB COMPOUND(S) ALIQUOT STD. CONC. STD. NO.			

DEQ; BOILER 5-26-94.

C4-131

MAY 05 '98 09:46 FR QUANTERRA 916 372 1059 TO 13606381685 P.63/66

PAGE # 96-_____

QUANTERRA ENVIRONMENTAL SERVICE, WEST SACRAMENTO

ENERGETICS AND SPECIALTY CHEMICALS GROUP (ESC)
STANDARD SOLUTION MADE FROM NEAT

SOLUTION ID#: _____

NAME OF STANDARD: _____

COMPONENT/SOURCE/STOCK#/LOT#/LOCATION/PURITY/EXP.	WEIGHT	FINAL CONC

(Additional components may be listed on back)

DESCRIPTION OF PREPARATION: BALANCE ID# _____

SPECIAL NOTES/COMMENTS:

ACCEPTABLE FOR USE? yes ___ no ___ QC'ed BY/DATE: _____

PREPARED BY/DATE: _____ REVIEWED BY/DATE: _____

STORAGE LOCATION: _____ EXPIRATION DATE: _____

DEG 2-1-96 / QA #359

PAGE # 96-_____

QUANTERRA ENVIRONMENTAL SERVICE, WEST SACRAMENTO

ENERGETICS AND SPECIALTY CHEMICALS GROUP (ESC)
STANDARD SOLUTION MADE FROM SOLUTION(S)

SOLUTION ID#: _____

NAME OF MIX: _____

COMPONENT/ID#/LOCATION/CONCENTRATION/SOLVENT/ EXPIRATION	VOLUME USED	FINAL CONC

(Additional components may be listed on back)

DESCRIPTION OF PREPARATION:

_____

_____

_____

SPECIAL NOTES/COMMENTS:

_____

_____

ACCEPTABLE FOR USE? yes ___ no ___ QC'ed BY/DATE: _____

PREPARED BY/DATE: _____ REVIEWED BY/DATE: _____

STORAGE LOCATION: _____ EXPIRATION DATE: _____

DEG 2-1-96 / QA #360

C4-133

Quanterra Services Environmental Services

QUANTERRA ENVIRONMENTAL SERVICES (West Sacramento)
SOP CHANGE FORM

SOP Number:	SAC-LC-0001
SOP Title:	
SOP Sections Affected by Change:	SECTION 13.3
Reason for Addition or Change:	EQUATION WAS TYPED INCORRECTLY
Change Effective From [Date]:	SEPT. 3, 1997
Change or Addition (Specific Section; Use additional sheets if necessary):	HPLC / UV: AMOUNT FOUND (ppb) = $\frac{A_x \cdot V_x}{CF_c \cdot W_s}$

Submitted by/Date: Dennis E. Gall / 9-3-97

Approved by:*

Technical Reviewer: [signature] Date 9/3/97

EH&S Signature: Robert Dow for Cheryl Gill Date 9/3/97

QA Signature: [signature] Date 9/3/97

Management Signature: [signature] Date 9-3-97

* Must be same signature authorities of SOP being revised.

C4-134

QUANTERRA INC. (West Sacramento)

STANDARD OPERATING PROCEDURE CHANGE FORM

SOP Number:	SAC-LM-0001, Revision 5.0, 07/08/96.
SOP Title:	Determination of Nitroaromatics, Nitramines, and Specialty Explosives in Water and Soil/Sediments by HPLC/UV and by LC/TSP/MS
SOP Sections Affected by Change:	8.5, 12.1.4, 12.2.4, 12.4.4, 12.6.9, and 12.8.3.
Reason for Addition or Change:	Adding PETN with 8330 analytes by HPLC/UV.
Change Effective From [Date]:	May 4, 1998
Change or Addition (Specific Section; use additional sheets if necessary.):	8.5 Analytical Curve for 8330 by HPLC/UV. PETN is added to the 8330 Mix between 40 ng/mL to 1,000 ng/mL. 12.1.4 PETN is added to the LCS and MS/SD, or to the DCS, at 250 ppb by adding 25 uL of the 100 ug/mL standard to 10 mL water for DAI method 12.2.4 PETN is added to the LCS and MS/SD, or to the DCS, at 162 ppb by adding 125 uL of the 100 ug/mL standard to 77 mL water for high level salting method. 12.4.4 PETN is added to the LCS and MS/SD, or to the DCS, at 6.5 ppb by adding 50 uL of the 100 ug/mL standard to 770 mL water for low level salting method. 12.6.9 PETN is added to the LCS and MS/SD, or to the DCS at 5.0 ppm by adding 100 uL of the 100 ug/mL standard to 2.0 gram soil, and the sample is prep as per 8330. 12.8.3 The gradient program is modified for the elution of PETN. At 12 minutes, the solvent composition is 60:5:35, and at 15 minutes the solvent composition is 59:4:37, which is held till the end of the run. After the nitrotolunes elutes, the UV detection is switched at approximately 20.8 minutes to 214 nm to detect PETN at approximately 22.0 minutes.

Submitted by/Date:

C4-135

Approved by:*

Technical Reviewer: Karla Shuelder *Date:* 5/4/98

EH&S Signature: *Date:*

QA Signature: *Date:*

Management Signature: *Date*

**Must be same signature authorities of SOP being revised.*

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C4-1369

Quanterra
Environmental Services

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SOP No: CORP-OP-0001SAC
Revision No: 3.1
Revision Date: ~~12/15/97~~ 9/4/98
Implementation Date:

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: **EXTRACTION AND CLEANUP OF ORGANIC COMPOUNDS FROM WATERS AND SOILS, BASED ON SW-846 3500 SERIES, 3600 SERIES, 8151A AND 600 SERIES METHODS.**

(SUPERSEDES: Revision 2)

Prepared by: Richard Burrows

Reviewed by: _____
Technical Specialist

Approved by: _____
Director, Quality Assurance or Technology

Approved by: _____
Director, Health and Safety

Approved by: _____
Management

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION....4
2. SUMMARY OF METHOD....4
3. DEFINITIONS....5
4. INTERFERENCES....5
5. SAFETY....5
6. EQUIPMENT AND SUPPLIES....6
7. REAGENTS AND STANDARDS....8
8. SAMPLE COLLECTION PRESERVATION AND STORAGE....11
9. QUALITY CONTROL....11
10. CALIBRATION AND STANDARDIZATION....13
11. PROCEDURE....13
12. DATA ANALYSIS AND CALCULATIONS....44
13. METHOD PERFORMANCE....44
14. POLLUTION PREVENTION....45
15. WASTE MANAGEMENT....45
16. REFERENCES....46
17. MISCELLANEOUS....46

List of Appendices:

Appendix A — Extraction Procedure for Chlorinated Acid Herbicides Based on Method 8151A....68

List of Tables

Table 1 — Liquid / liquid extraction conditions
Table 2 — Exchange solvents and final volumes
Table 3 — Surrogate spiking solutions
Table 4 — Matrix spike and LCS solutions

Table 5	Surrogate spike components
Table 6	Matrix spike components
Table A1	Herbicide surrogate spiking solutions
Table A2	Herbicide matrix spike and LCS solutions
Table A3	Herbicide surrogate spike components
Table A4	Herbicide matrix spike and LCS components

1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, TCLP leachate, and soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS). The procedures are based on SW-846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8082, 8015, 8141A, 8151B, 8270C, 8310 608, 610, and 625. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.1. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Continuous Liquid/Liquid Extraction

A measured volume of sample, typically 1 liter, is placed into a continuous liquid/liquid extractor, adjusted, if necessary, to a specific pH and extracted with methylene chloride for 18-24 hours.

2.3. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using an ultrasonic horn.

2.3.1. **Hearing protection** is required when sonicating samples.

2.4. Soxhlet Extraction

A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent for 16-24 hours.

2.5. Cleanup and Concentration

Procedures are presented for removing interferents from sample extracts, and for drying and concentration of the extract to final volume for analysis.

2.6. Phenoxy acid herbicide extractions

Procedures for the extraction and cleanup of phenoxy acid herbicides are presented in Appendix A.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates. The following requirements must be met:

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and *chemically resistant* gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded. N-Dex Plus nitrile gloves may be used when these solvents are handled. While these gloves provide good intermittant splash protection, they give little or no protection against contact with large quantities of solvent, and no protection against spills or immersion.

5.2.1. Laminate gloves (4H®, Silver Shield® with a nitrile second glove) provide the best protection against dichloromethane and provide a wide range of protection against other chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure

have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.3.1. Chemicals that have been classified as **carcinogens** or **potential carcinogens** under OSHA include methylene chloride, polychlorinated biphenyls (PCBs), certain pesticides.

5.3.2. The following materials are known to be **corrosive**: Concentrated sulfuric acid, sodium hydroxide, potassium hydroxide, nitric acid.

5.3.2.1. Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

5.3.3. Chemicals known to be **flammable** are: Diethyl ether, acetone, hexane, 2-propanol, *acetonitrile*.

5.3.4. *Chemicals known to be **neurotoxic agents** are: hexane.*

5.3.5. *Chemicals known to readily absorb via the skin are: acetonitrile, mercury.*

5.4. Mercury is a highly toxic compound that must be handled with care. The analyst must be aware of the appropriate handling and clean-up techniques before handling this material. ***DO NOT** use latex gloves when working with mercury.*

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit. Use of methylene chloride for glassware cleaning should be avoided.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned with soap and water, rinsed with water and dried in

an oven at 400°C for at least 2 hours. Alternatively the glassware can be solvent rinsed with acetone or methanol followed by methylene chloride after the water rinse. *Heat resistant gloves must be used when handling kilned glassware.*

6.2. Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Separatory Funnel: 2 L	√				
Separatory Funnel Rack	√				
Balance: >1400 g capacity, accurate ±1 g	√	√			
pH indicator paper, wide-range: covers extraction pH	√	√			
Graduated cylinder: 1 liter. (other sizes may be used)	√	√			
Erlenmeyer Flask or Fleaker: 125 & 300 mL (other sizes optional)	√		√		
Solvent Dispenser Pump or 100 mL Graduated Cylinder	√		√		
Continuous Liquid/Liquid Extractor		√			
Round or flat Bottom: 250, 500 mL or 1 L		√			
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).		√		√	√
Cooling Condensers		√		√	
Heating Mantle: Rheostat controlled		√		√	
Auto-timer for heating mantle		√		√	
Beakers: 250 & 400 mL, graduated			√		
Balance: >100 g capacity, accurate ±0.1 g			√	√	
Soxhlet Extractor				√	
Sonicator (at least 300 watts)			√		
Sonicator horn, 3/4 inch			√		
Kuderna-Danish (K-D) Apparatus: 500 mL					√
Concentrator Tube: 10 mL, attached to K-D with clips					√
Snyder Column: Three-ball macro					√
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.					√
Vials: Glass, 2 mL, 4 mL, and 10 mL capacity with Teflon®-lined screw-cap					√
Nitrogen Blowdown Apparatus					√
Nitrogen: reagent grade.					√
Culture tubes: 10 mL, 16 mmx100 mm					√
Syringe: 1 mL	√	√	√	√	
Phase Separation Paper	√	√	√	√	
Glass Wool	√	√	√	√	
Glass Funnel: 75 X 75 mm	√	√	√	√	√
Disposable Pipets	√	√	√	√	√
Aluminum foil	√	√	√	√	√

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Paper Towels	√	√	√	√	√

6.3. Equipment and Supplies for Cleanup Procedures

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid	A/B
Gel permeation chromatography system (GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc.. or Zymark Benchmate or equivalent).	√				
Bio Beads: (S-X3) -200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent).	√				
Chromatographic column: 700 mm x 25 mm ID glass column. Flow is upward.	√				
Ultraviolet detector: Fixed wavelength (254 nm) and a semi-prep flow-through cell.	√				
Strip chart recorder, recording integrator, or laboratory data system.	√				
Syringe: 10 mL with Luerlok fitting.	√				
Syringe filter assembly, with disposable 5 um filter discs, Millipore No. LSWP 01300 or equivalent.	√				
Chromatographic column: 250 mm long x 10 mm ID; with Pyrex glass wool at the bottom and a Teflon stopcock (for silica gel cleanup).	√				
Vacuum system for eluting multiple cleanup cartridges. Vac Elute Manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.		√			
Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.		√			
Vacuum pressure gauge.		√			
Rack for holding 10 mL volumetric flasks in the manifold.		√			
Mechanical shaker or mixer: Vortex Genie or equivalent.			√	√	
Separatory Funnels with Ground-Glass Stoppers: 250 mL					√
Erlenmeyer Flasks: 125 mL					√
Disposable Pipets		√	√	√	√
Culture tubes: 10 mL, 16 mmx100 mm	√	√	√	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep fun.	CLLE	Soni	Sox	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	√	√			
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	√			
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	√	√			
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	√			
Organic free reagent water.	√	√			
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√	√	√	
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√	√	√	√
Acetone: Used for cleaning	√	√	√	√	√

7.2. Reagents for Cleanup Procedures

REAGENTS	GPC	Florisil	Sulfur	Acid	A/B
Florisil: 500 mg or 1 g cartridges with stainless steel or Teflon frits (calalog 694-313, Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent.)		√			
Mercury: triple distilled			√		
Tetrabutylammonium hydrogen sulfate			√		
Sodium sulfite			√		
Tetrabutylammonium (TBA) sulfite reagent: Prepare reagent by dissolving 3.39 g of Tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. Extract this solution 3 times with 20 mL portions of hexane. Discard the hexane extracts. Add 25 g sodium sulfite to the water solution.			√		
2-Propanol			√		
Nitric acid: 1N			√		
Copper powder: remove oxides (if powder is dark) by treating with 1N nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.			√		
Sulfuric acid, Concentrated				√	
Sodium hydroxide, Pellets					√
Sodium hydroxide, 10N: Dissolve 40 g of NaOH in 100 mL of reagent water					√
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade					√
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.					√

7.3. Standards

7.3.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from neats. Semivolatile stock standards are stored at $\leq 6^{\circ}C$. All stock standards must be protected from light.

7.3.1.1. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased.) Standards must be allowed to come to room temperature before use.

7.3.1.2. Expired standards must be rotated out of the lab to the Hazardous Waste storage area.

7.3.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.4. GPC calibration solution - prepare or purchase a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/mL
Corn Oil	25.0
Bis (2-ethylhexyl) phthalate	1.0
Methoxychlor	0.2
Perylene	0.02
Sulfur	0.08

NOTE: Sulfur is not very soluble in methylene chloride, however, it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds. This

standard has a lifetime of 6 months.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2. For TCLP leachates, extraction is initiated within seven days from when the leaching procedure is completed.

8.3.3. Analysis of the extracts is completed within forty days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See policy QA-003 for further definition of the batch.

9.2. Definition of matrix

The possible matrix types are aqueous, soil, waste and TCLP leachate.

9.3. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Use of a LCS pair in place of a MS/MSD must be documented.

9.4. Sample count

Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.5. Method Blank

A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background

interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1. Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2. Solid method blanks use 30 g of sodium sulfate spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.3. TCLP method blanks use 200 mL of leachate fluid (100 mL for pesticides or herbicides) spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.6. Laboratory Control Sample (LCS)

Laboratory Control Samples are well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1. The LCS is made up in the same way as the method blank (See sections 9.5.1 - 9.5.3) for water samples but spiked with the LCS standard and the surrogates.

9.6.2. A solid LCS uses 30g of sodium sulfate and 30g of reagent sand, then spiked with the LCS standard and the surrogates.

9.7. Surrogates

9.7.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2. Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike.

9.9. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in section 13 must be acceptable before analysis of samples may begin.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.11. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. On a daily basis, measure 1.0 mL of solvent into an autovial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL (1 mL total volume). The "standard" autovial is sealed and the top and bottom of the meniscus are marked. The autovials containing the sample extracts are then compared against the "standard" vial to ensure that the final volume is consistently 1.0 ± 0.01 mL. If a new box of autovials are used, then the steps are repeated to further ensure that variations due to vial size and shape are minimized.

10.2. Refer to section 11.8.1.2 for calibration of the GPC. Otherwise this section is not applicable.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (11.2), continuous liquid/liquid extraction (11.3), sonication extraction (11.4), soxhlet extraction (11.5), waste dilution (11.6), extract concentration (11.7), and extract cleanup (11.8) are presented in this section.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a

supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples.

A flow chart for this procedure is included in Section 17.

11.2.1. For pesticides and TCLP leachates, measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.2.2. The normal sample volume is 1 liter. Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples.

11.2.3. Weigh the sample container on a balance (± 1 g), taring the sample and container. Add the surrogate spiking solution. Also add matrix spiking solution to any matrix spike / matrix spike duplicate samples. Mix well. Transfer the sample to the separatory funnel. Rinse the sample bottle with 60 mL methylene chloride and transfer to the separatory funnel. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer the sample to the separatory funnel and then add the spike.

Warning: **Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately** after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.3.1.If the entire sample bottle will not be used, transfer the aliquot to the separatory funnel, then add the spiking solutions to the sample in the separatory funnel.

Note: Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be

necessary for specific client programs.

11.2.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The LCS is spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates.

11.2.5. Use 100 mL of leachate for TCLP pesticides, and 200 mL of leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate may be made up to 1 L in volume with reagent water.

11.2.6. For a TCLP method blank, measure 100 mL (200 mL for semivolatiles) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction if desired.

11.2.7. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.2.8. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

Warning: **Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately** after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.9. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (approximately 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction (Section 11.3.). If this is done, the sample must be extracted as part of a valid CLLE batch.

**Note:* 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

11.2.10. Fill a funnel with 10-20 g of anhydrous sodium sulfate. The funnel can be plugged with glass wool or filter paper may be used to hold the sodium sulfate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

11.2.11. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.12. If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride, as outlined in Steps 11.2.7 to 11.2.9. Collect these three extracts in the same container used for the acid fraction.

Note: Alternatively, the acid and base fractions may be kept separate. This may be required for method 625. Separate analysis of the acid and base fractions may also be required for method 625. Individual client requirements must be checked before starting the extraction.

11.2.13. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.14. Dispose of solvent and water remaining in the extractor into the LLE waste drum. Waste methylene chloride goes to the DCM drum for recycling.

11.2.15. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.3. Continuous Liquid/Liquid Extraction from Water Samples.

A flow chart for this procedure is included in Section 17.

11.3.1. Assemble the apparatus. Add 300-500 mL of methylene chloride to the

extractor body. Add 3 to 5 boiling chips to the round-bottom distilling flask.

11.3.2. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.3.3. Weigh the sample container on a balance (± 1 g), taring the sample and container. Add the surrogate spiking solution. Also add matrix spiking solution to any matrix spike / matrix spike duplicate samples. Mix well. Transfer the sample to the extractor. Rinse the sample bottle with 60 mL methylene chloride and transfer to the extractor. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer the sample to the extractor and then add the spike.

Warning: **Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately** after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

11.3.3.1.If the entire sample bottle will not be used, transfer the aliquot to the extractor, then add the spiking solutions to the sample in the extractor.

Note: Alternative methods of measurement of sample volume include: a.) transferring the sample to a measuring cylinder and b.) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.

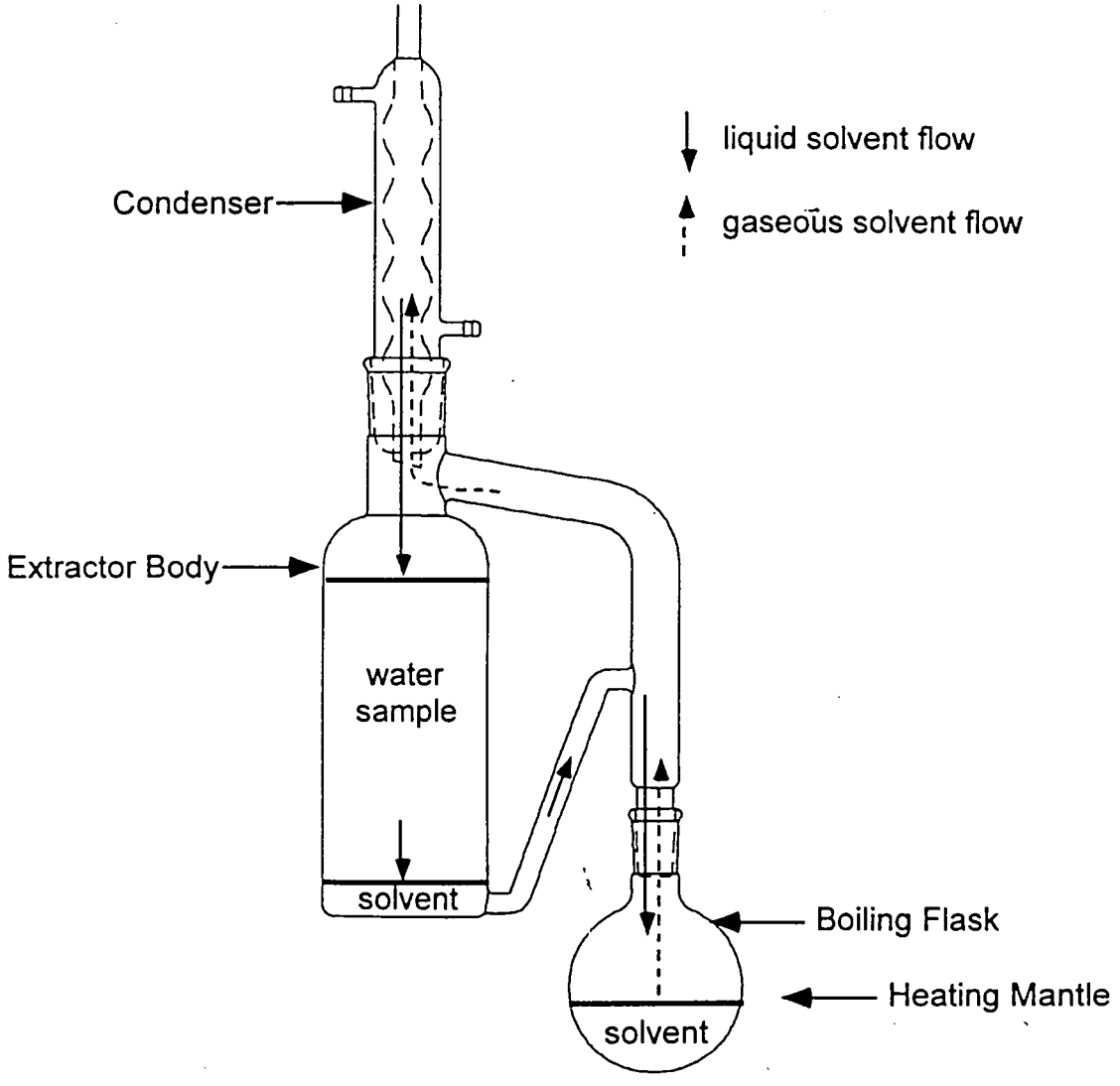
11.3.3.2.Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The method blank is spiked with the surrogates, the LCS and matrix spikes with the surrogates and matrix spiking solutions. Note that different spiking solutions are used for methods 625, 8270 and TCLP.

11.3.3.3.Use 100 mL of leachate for TCLP pesticides, and 200 mL of

leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate may be made up to 1 L in volume with reagent water.

11.3.3.4.For a TCLP method blank, measure 100 mL (200 mL for semivolatiles) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water.

11.3.3.5.Less than one liter of sample may be used, for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event dilute the sample to about 1 liter with reagent water.



11.3.4. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the

sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.3.5. Add reagent water to the extractor body until approximately 250 mL of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Attach cold condenser (about 12°C). Turn on heating mantle. **Inspect joints for leaks** once solvent has begun cycling. Extract for 18-24 hours. (24 hours required for 600 series)

11.3.6. If extraction at a secondary pH is required, (see Table 1) turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. If desired, the acid and base fractions may be kept separate by replacing the boiling flask with a clean flask and fresh solvent. Reattach the condenser and turn on heating mantle. Extract for 18-24 hours.

Note: Alternatively, the acid and base fractions may be kept separate. This may be required for method 625. Separate analysis of the acid and base fractions may also be required for method 625. Individual client requirements must be checked before starting the extraction.

11.3.7. Turn off the heating mantle and allow the extractor to cool.

11.3.8. Place a funnel containing 10-20 g of anhydrous sodium sulfate on the Kuderna-Danish (K-D) apparatus or other glass container. The funnel can be plugged with glass wool enabling it to hold the granular anhydrous sodium sulfate or phase separation filter paper may be used.

11.3.9. Dry the extract in the round bottom flask by filtering it through the sodium sulfate filled funnel. Note that it is not necessary or advisable to attempt to add the solvent remaining in the continuous extractor body to the extract.

11.3.10. Collect the dried extract in a K-D or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer. Dispose of solvent and water remaining in the extractor in the *LLE waste drum. Waste methylene chloride goes to the DCM drum for recycling.*

Note: Some types of CLLE apparatus have built in drying columns. If this type of apparatus is used then a drying step subsequent to the extraction may not be necessary.

11.3.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4. Sonication

A flow chart for this procedure is included in Section 17.

11.4.1. Determination of percent moisture (Optional - if a different group performs this test, refer to the facility SOP.)

11.4.1.1. In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight (at least 12 hours) at 110 °C ± 5°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.4.2. Determination of pH (Optional - if a different group performs this test, refer to the facility SOP.)

11.4.2.1. If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for five minutes then let stand for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.4.3. Decant and discard any water layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator). Document if a water layer was discarded.

11.4.4. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.4.5. Weigh 30 g of sample ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0. 1 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate for the method blank and 30 g of sodium sulfate with 30 g of reagent sand for the LCS.

11.4.6. Mix weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.7. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP.

11.4.8. Add the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add 1 the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogate and matrix spiking solution is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

11.4.9. Immediately add a minimum of 100 mL of solvent to the beaker.

Analysis	*Solvents*
Semivolatile GC/MS, OPP, PAH by HPLC	*1:1 Methylene Chloride/Acetone*
Organochlorine pesticides and PCBs	*1:1 Hexane / Acetone*
Hydrocarbons by FID (DRO, etc.)	*Methylene Chloride*

Note: Steps 11.4.5 - 11.4.9 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.10. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.11. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn. with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: Do ***not*** use ***Microtip*** probe. **Hearing protection is required when sonicating samples.**

11.4.12. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.4.13. Place the prepared funnel on a collection apparatus (beaker or K-D Apparatus.

11.4.14. Decant and filter extracts through the prepared funnel into a clean beaker or K-D Apparatus.

11.4.15. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.4.16. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4.17. Sonicator Tuning.

11.4.17.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed.

11.5. Soxhlet

11.5.1. Determination of % moisture

11.5.1.1. In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.5.2. Determination of pH

11.5.2.1. If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.5.3. Decant and discard any water layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as

sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client. Document on benchsheet if a water layer was discarded.

11.5.4. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.5.5. Weigh 30 g of sample ± 1.0 g into a beaker or glass jar, recording the weight to the nearest 0.1 g on the benchsheet.. Use 30 g of sodium sulfate for the method blank and 30g of sodium sulfate and 30g of reagent sand for the LCS. Add 30 g of anhydrous sodium sulfate to the samples and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.5.5.1.Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.

11.5.6. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.5.7. Add the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 - 6 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogates and matrix spiking compounds is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

11.5.8. Place approximately 300 mL of solvent into a 500 mL flat bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Analysis	*Solvents*
Semivolatile GC/MS, OPP, PAH by HPLC	*1:1 Methylene Chloride / Acetone*
Organochlorine pesticides and PCBs	*1:1 Hexane / Acetone*
Hydrocarbons by FID (DRO, etc.)	*Methylene Chloride*

11.5.9. Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask. Dry the extract in the flask by filtering it through a sodium sulfate filled funnel.

11.5.10. Collect the dried extract in a K-D or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer.

11.5.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.6. Waste Dilution

11.6.1. This method is used for materials that are soluble in an organic solvent.

11.6.2. Transfer 10 mL of the solvent to be used for dilution into a Teflon capped vial. Mark the meniscus on the vial, then discard the solvent.

11.6.3. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest 0.1 g.

11.6.4. Add the surrogate solution (Table 3) For GC/MS analysis, the surrogate solution should be 10x more concentrated than the normal solution (i.e. 1000/1500 ppm). For pesticides the normal surrogate solution is appropriate.

11.6.5. Dilute to 10 mL with the appropriate solvent. (Methylene Chloride for GC/MS analysis, hexane for pesticides analysis.)

11.6.6. Add 2 g + 0.1 g sodium sulfate to the sample. Cap and shake for 2 minutes.

11.6.7. Add 4-5 g sodium sulfate to a small funnel. The funnel can be plugged

with glass wool or phase separation filter paper may be used to hold the sodium sulfate.

11.6.8. Pour the sample through the funnel, collecting as much as possible in a clean vial. Do NOT rinse the funnel with additional solvent, and do NOT concentrate the sample. The final volume is defined as 10 mL.

11.6.9. Label the sample, which is now ready for cleanup or analysis.

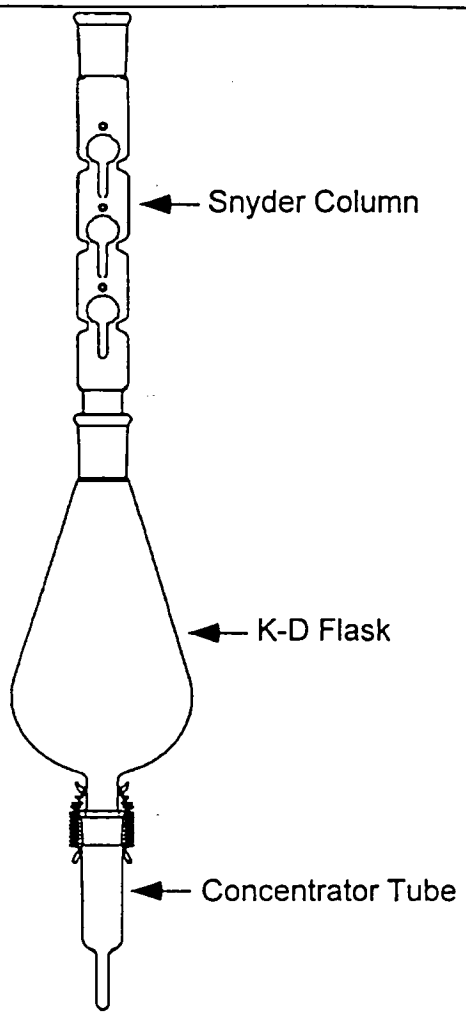
11.6.10. Add 10ml of the solvent used for the waste dilution to a test tube, label as the Method Blank.

11.7. Concentration

According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.7.1. Kuderna-Danish (KD) Method:

11.7.1.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Transfer the sample to the K-D flask.Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly).



11.7.1.2.Place the KD apparatus on a water bath (80-90°C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

11.7.1.3.Concentrate to 5-15 mL. If the determinative method requires a solvent exchange add the appropriate exchange solvent (50 mL hexane, 50 mL cyclohexane, 2 mL acetonitrile or 10 mL toluene) to the top of the Snyder Column, and then continue the water bath concentration back down to 1-4 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: Add an additional boiling chip with the addition of exchange solvent.

An alternative technique for solvent exchange is to replace the macro Snyder column and KD flask with a micro Snyder column, concentrate to approximately 1 mL, add 10 mL of hexane, and concentrate back down to 1 mL. The extract must be cool before the macro Snyder assembly is removed.

Note: It is very important not to concentrate to dryness as analytes will be lost *and an unsafe condition will be created.*

11.7.1.4.Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.7.1.5.If the final volume is 5 or 10 mL the extract may be made up to volume in the graduated KD tube or transferred to a 12 mL vial previously marked at the appropriate volume level. Document the final volume. Otherwise proceed to section 11.7.2

11.7.2. Nitrogen Evaporation to Final Concentration

11.7.2.1.Transfer the entire extract to a calibrated evaporation tube. Rinse the concentrator tube with 1-2 mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

11.7.2.2.Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

11.7.2.3.During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent accurately to the calibrated volume line

and transfer the extract to the appropriate storage vial.

Note: It is very important **not to concentrate to dryness** as analytes will be lost *and an unsafe condition will be created.*

11.7.2.4.An alternative technique is to follow the previous steps concentrating the solvent to slightly below the required final volume and then drawing the extract into a syringe. Rinse the evaporation tube with a small amount of solvent and draw additional solvent into the syringe to make up the accurate final volume.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.7.3. Turbovap Method

11.7.3.1.Turn on the Turbovap and adjust the water temperature to 5-10°C less than the boiling point of the solvent to be evaporated.

11.7.3.2.Switch all endpoint sensors to the "NO" position.

11.7.3.3.Adjust the water bath level

11.7.3.4.Adjust the nitrogen gas pressure to approximately 12 psi.

11.7.3.5.Transfer the extract into the Turbovap tube and load into the Turbovap. Do not fill the Turbovap tubes over approximately 3/4 full.

11.7.3.6.Reset the sensor and close the lid.

Note: If the extract splashes when the nitrogen flow starts, reduce the nitrogen flow or transfer a portion of the extract back into the original extract container.

11.7.3.7.As the extract concentrates, transfer the remainder of the extract into the appropriate Turbovap tube. After all of the extract has been transferred, rinse the flask with a few mL of methylene chloride and add to the Turbovap tube.

11.7.3.8.During the concentration rinse the Turbovap tube walls with a few mL of solvent 1 or 2 times.

11.7.3.9. Concentrate the extract to slightly less than the required final volume.

11.7.3.10. If solvent exchange is required, concentrate to 1-4 mL and add 50 mL of the exchange solvent. Concentrate back down to the appropriate volume. Refer to table 2 for details of exchange solvents and final volumes.

11.7.3.11. Transfer the concentrated extract to volumetric glassware for adjustment of final volume, using a small amount of solvent to rinse the tube and complete the transfer.

Note: Water contamination from condensation during concentration is not acceptable. If water is present, remove the Turbovap tube and filter the extract through sodium sulfate. Transfer to a clean Turbovap tube and continue the concentration.

Note 2: Dark, opaque or turbid samples may not concentrate. If this occurs, set the concentrator sensor to dryness and supervise the entire concentration procedure.

11.8. Cleanup Techniques

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/ or loss of detector sensitivity.

Gel Permeation Chromatography (Section 11.8.1) is a generally applicable technique which can be used to prepare extracts for Semivolatiles (8270), PAH (8100) and pesticides (8080) analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.

Florisil column cleanup (Section 11.8.2) is particularly useful for cleanup of pesticides for analysis by method *8081A* and should normally be applied to these samples unless the matrix is clean. It separates compounds with a different polarity from the target analytes.

Gel Permeation Chromatography and Florisil column cleanup may both be applied to samples for analysis by method *8081A*. In this case the GPC should be performed first.

Sulfur cleanup (Section 11.8.3) is generally applied to samples for analysis by method *8081A*, since the Electron Capture Detector responds strongly to sulfur. It is performed after GPC and Florisil cleanup.

Sulfuric acid cleanup (Section 11.8.4) is applied to samples requiring analysis for

8082 Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by the sulfuric acid.

WARNING: Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

Acid Base Partition Cleanup (Section 11.8.5) is useful for separating organic acids and phenols from basic and neutral organics.

Silica gel cleanup (Section 11.8.6) may be used to separate polynuclear aromatic hydrocarbons from interfering aliphatic hydrocarbons prior to analysis by GC/FID.

11.8.1. Gel Permeation Chromatography (GPC)

Note: GPC systems include the GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc., Zymark Benchmate, or equivalent.

11.8.1.1.GPC Column Preparation

11.8.1.1.1.Weigh out 70 g of Bio Beads (SX-3) into a 400-mL beaker.

11.8.1.1.2.Add approximately 300 mL of methylene chloride and stir gently.

11.8.1.1.3.Cover with aluminum foil and allow the beads to swell for a minimum of two hours. Maintain enough solvent to sufficiently cover the beads at all times.

11.8.1.1.4.Position and tighten the outlet bed support (top) plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger near the column end but no closer than 5 cm (measured from the gel packing to the collar).

11.8.1.1.5.Turn the column upside down from its normal position with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.

11.8.1.1.6.Swirl the bead/solvent slurry to get a homogeneous mixture and pour the mixture into the open end of the column. Transfer as much as possible with one continuous pour trying to minimize bubble formation. Pour enough to fill the column. Wait for the excess solvent to drain out

before pouring in the rest. Add additional methylene chloride to transfer the remaining beads and to rinse the beaker and the sides of the column. If the top of the gel begins to look dry, add more methylene chloride to rewet the beads.

11.8.1.1.7.Wipe any remaining beads and solvent from the inner walls of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

11.8.1.1.8.Push the plunger until it meets the gel, then compress the column bed about 4 cm.

11.8.1.1.9.Connect the column inlet to the solvent reservoir and place the column outlet tube in a waste container. Pump methylene chloride through the column at a rate of 5 mL/min. for one hour.

11.8.1.1.10.After washing the column for at least one hour, connect the column outlet tube to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. Placing a restrictor (made from a piece of capillary tubing of 1/16"OD x 10/1000"ID x 2") in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect the flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi back-pressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

11.8.1.1.11.When the GPC column is not to be used for several days, connect the column inlet and outlet lines to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, pump methylene chloride through the column until the observed

column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

11.8.1.2.Initial Calibration of the GPC Column

11.8.1.2.1.Before use, the GPC must be calibrated based on monitoring the elution of standards with a UV detector connected to the GPC column.

11.8.1.2.2.Pump solvent through the GPC column for 2 hours. Verify that the flow rate is 4.5-5.5 mL/min. Corrective action must be taken if the flow rate is outside this range. Record the column pressure (should be 6-10 psi) and room temperature (22°C is ideal).

Note: Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared.

11.8.1.2.3.Inject the calibration solution and retain a UV trace that meets the following requirements (See resolution calculation in section 11.8.1.7):

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

11.8.1.2.4.A UV trace that does not meet the criteria in paragraph 11.8.1.2.3 indicates the need for system maintenance and/or the need for a new column.

11.8.1.2.5.Determine appropriate collect and dump cycles.

11.8.1.2.6.The calibrated GPC program for pesticides/PCB should dump >85% of the phthalate and should collect >95% of the methoxychlor and perylene. Use a wash time of 10 minutes.

11.8.1.2.7.For semivolatile extracts, initiate a column eluate collection just before the elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Stop collection before sulfur elutes. Use a wash time of 10 minutes after the elution of sulfur.

11.8.1.2.8.Reinject the calibration solution after appropriate dump and collect cycles have been set.

11.8.1.2.9.Measure and record the volume of collected GPC eluate in a graduated cylinder.

11.8.1.2.10.The retention times for both bis(2-ethylhexyl) phthalate and perylene must not vary more than +/- 5% between calibrations.

11.8.1.3.GPC calibration check

Check the calibration of the GPC immediately after the initial calibration and at least every 7 days thereafter, while the column is in use.

11.8.1.3.1.Inject the calibration solution, and obtain a UV trace. If the retention times of bis(2-ethylhexyl)phthalate or perylene have changed by more than ± 5% use this run as the start of a new initial calibration. Otherwise, proceed with the recovery check. Excessive retention time shifts may be caused by poor laboratory temperature control or system leaks, an unstabilized column, or high laboratory temperature causing outgassing of methylene chloride. Pump methylene chloride through the system and check the retention times each day until stabilized.

11.8.1.4.GPC Recovery Check for Pesticides/ PCBs

11.8.1.4.1.The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Two recovery check solutions are used. The first mixture is prepared by diluting 1.0 mL of the pesticide matrix spiking solution (Table 6) to 10 mL in methylene chloride. The second mixture is prepared by diluting 1 mL of the PCB only matrix spiking solution (Table 6) to 10 mL with methylene chloride.

11.8.1.4.2.Load the pesticide matrix spike mixture, the PCB mixture, and a methylene chloride blank onto the GPC using the GC dump and collect values.

Note: If the analysis is for PCBs only, then the pesticide recovery check is not necessary.

11.8.1.4.3.After collecting the GPC calibration check fraction, concentrate, solvent exchanging to hexane. Adjust the final volume to 5.0 mL, and analyze by GC/EC. Refer to concentration, section 11.7.

11.8.1.4.4.The methylene chloride blank may not exceed more than one half the reporting limit of any analyte. And if the recovery of each of the single component analytes is 80-110% and if the Aroclor pattern is the same as previously run standards, then the analyst may use the column. If the above criteria are not met, there may be a need for system maintenance.

11.8.1.5.GPC Recovery Check for Semivolatiles

11.8.1.5.1.The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Dilute 1.0 mL of the semivolatiles matrix spiking solution (Table 6) to 10 mL in methylene chloride.

11.8.1.5.2.Load the matrix spike mixture and a methylene chloride blank onto the GPC using the semivolatiles dump and collect values.

11.8.1.5.3.After collecting the GPC recovery check fraction, concentrate to 0.5 mL, and analyze by GC/MS. Refer to the concentration section 11.7.

11.8.1.5.4.Recovery of the matrix spike analytes should be at least 60%. The blank should not contain any analytes at or above the reporting limit. If these conditions are met the column may be used for sample analysis. Otherwise correct the contamination problem, or extend the collect time to improve recovery of target analytes.

11.8.1.6.Sample Extract Cleanup

11.8.1.6.1.Reduce the sample extract volume to 1-2 mL, then adjust to 10 mL with methylene chloride prior to cleanup. This reduces the amount of acetone in the extract. Refer to section 11.7.

11.8.1.6.2.Start the pump and let the flow stabilize for 2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C. The normal backpressure is 6-10 psi.

11.8.1.6.3.In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution (by visual comparison) must be diluted and loaded into several loops.

11.8.1.6.4.Samples being loaded onto the GPC should be filtered with a 5 micron (or less) filter disk. Attach a filter to a 10 mL Luerlok syringe and filter the 10 mL sample extract into the sample tube.

Note: The Zymark Benchmate automatically filters the sample extracts, but manual prefiltration of very dirty extracts may be necessary.

11.8.1.6.5.Load the filtered (or unfiltered if using the Benchmate) samples into the proper sample tubes and place on the GPC.

Note: For the GPC Autoprep Model 1002A, wash the loading port with methylene chloride after loading each sample loop in order to minimize cross contamination. This step is automated on the GPC Autoprep 1002B and on the Benchmate.

11.8.1.6.6.Set the collect, dump, and wash times determined by the calibration procedure.

11.8.1.6.7.Switch to the run mode and start the automated sequence. Process each sample using the collect and dump cycle times established by the calibration procedure.

11.8.1.6.8.Collect each sample in a suitable glass container. Monitor sample volumes collected.

11.8.1.6.9.Any samples that were loaded into 2 or more positions must be recombined.

11.8.1.6.10.Concentrate semivolatile sample extracts to 0.5 mL in methylene chloride. Refer to the concentration section 11.7.

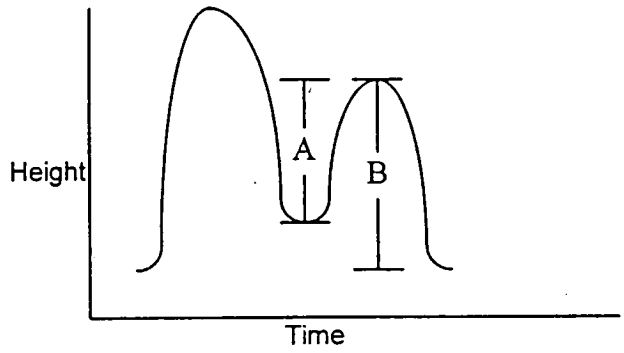
11.8.1.6.11.Solvent exchange pesticide/PCB sample extracts into hexane and concentrate to 5.0 mL. Refer to the concentration section 11.7.

11.8.1.7.Calculations

11.8.1.7.1.Resolution

To calculate the resolution between two peaks on a chromatograph, divide the depth of the valley between the peaks by the peak height of the smaller peak being resolved and multiply by 100.

Resolution Calculation



$$\% \text{ Resolution} = \frac{A}{B} \times 100$$

Where: A = depth of valley to height of smaller peak
B = peak height of smaller peak

11.8.1.7.2.Dump Time

Mark on the chromatograph the point where collection is to begin. Measure the distance from the injection point. Divide the distance by

the chart speed. Alternatively the collect and dump times may be measured by means of an integrator or data system.

$$\text{Dump time (min)} = \frac{\text{Distance (cm) from injection to collection start}}{\text{Chart speed (cm / min)}}$$

11.8.1.7.3.Collection Time

$$\text{Collection time (min)} = \frac{\text{Distance (cm) between collection start and stop}}{\text{Chart speed (cm / min)}}$$

11.8.2. Florisil Cartridge Cleanup

Florisil cleanup is generally used for organochlorine pesticides, although it may be applied to other analytes. Sections 11.8.2.1 through 11.8.2.8 outline the procedure for organochlorine pesticides, while section 11.8.2.9 outlines modifications required for other analytes.

Note 1: Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assembly, Zymark Benchmate, or equivalent.

Note 2: Follow the lab specific procedure when using the Zymark benchmate.

11.8.2.1.Before Florisil cleanup sample volume must be reduced to 10 mL (5 mL if GPC cleanup was used) and the solvent must be hexane. Refer to Section 11.7 for details of concentration.

11.8.2.2.Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.8.2.3.Place one Florisil cartridge into the vacuum manifold for each sample extract. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of hexane/acetone (9:1). Adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min. Do not allow the cartridges to go dry.

11.8.2.4.Just before the cartridges go dry, release the vacuum to the manifold and remove the manifold top.

11.8.2.5.Place a rack of clean labeled 12 mL concentrator tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.8.2.6.After the clean tubes are in place, vacuum to the manifold is restored and 2.0 mL of the extract is added to the appropriate Florisil cartridge.

11.8.2.7.The pesticides/aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 10 mL culture tube or concentrator tube held in the rack inside the vacuum manifold.

11.8.2.8.Transfer the extract to a graduated concentrator tube and concentrate the extract to 2 mL. Refer to the concentration section. (11.7)

Note 1: A cartridge performance standard must be run with each lot of Florisil cartridges.

Note 2: Florisil cartridge performance check--every lot number of Florisil must be tested before use. Add 0.5 ug/mL of 2,4,5-trichlorophenol solution and 0.5 mL of "*GC Organochlorine Standard Mix* A" (midpoint concentration) to 4 mL hexane. Reduce volume to 0.5 mL. Add the concentrate to a pre-washed Florisil cartridge and elute with 9 mL hexane/acetone [(90:10)(v/v)]. Rinse cartridge with 1.0 mL hexane two additional times. Concentrate eluate to 1.0 mL final volume and transfer to vial. Analyze the solution by GC/EC. The test sample must show 80 to 120% recovery of all pesticide analytes with <5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. This standard has a lifetime of six months. Alternatively, this standard may be purchased as a stock solution. *Rotate expired standards out of the laboratory for Hazardous Waste disposal.*

11.8.2.9.Modifications for other analyte classes

11.8.2.9.1.PCBs

Pre-elute the cartridge with 4 mL hexane. Add 2 mL of the sample extract and elute with 3 mL hexane. The eluant will contain the PCBs together with any heptachlor, aldrin, 4,4'DDE and part of any 4,4'DDT. Any BHC isomers, heptachlor epoxide, chlordane, endosulfan I and II, endrin aldehyde and endrin sulfate and methoxychlor will be retained on the column and can be eluted in a

separate fraction with 8 mL 90:10 hexane:acetone if required.

11.8.3. Sulfur Removal

11.8.3.1.Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA) according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipet, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

11.8.3.2.Sulfur Removal with Elemental Mercury

Note: Use Mercury sparingly in order to minimize exposure and disposal costs.

11.8.3.2.1.Transfer 2 mL of sample extract into a clean concentrator tube or Teflon sealed vial.

11.8.3.2.2.Add one to three drops of mercury to the extract vial and seal.

11.8.3.2.3.Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

11.8.3.2.4.Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

11.8.3.2.5.If black precipitate forms, sulfur was present. Shake again, then centrifuge. After centrifugation, transfer the supernate to a clean test tube and repeat. Do this until relatively little precipitate remains, or the screens indicate that cleanup is complete.

11.8.3.2.6.*Rotate the mercury waste out of the laboratory to the Hazardous Waste storage area for labpack disposal.*

11.8.3.3.Sulfur Removal with Copper

11.8.3.3.1.Transfer 1.0 mL of sample extract into a centrifuge or concentrator tube.

11.8.3.3.2.Add approximately 2 g of cleaned copper powder (see 7.2 for copper cleaning procedure) to the sample extract tube.

11.8.3.3.3.Mix for one minute on a mechanical shaker.

11.8.3.3.4.If the copper changes color, sulfur was present. Repeat the sulfur removal procedure until the copper remains shiny.

11.8.3.3.5.Transfer the supernate to a clean vial.

11.8.3.4.Sulfur Removal with Tetrabutylammonium (TBA) Sulfite Reagent

11.8.3.4.1.Transfer 1.0 mL of sample extract into a culture tube.

11.8.3.4.2.Add 1.0 mL TBA sulfite reagent and 2 mL 2-propanol to the sample extract. Cap and shake for 1 minute. If clear crystals (precipitated sodium sulfite) form, sufficient sodium sulfite is present.

11.8.3.4.3.If a precipitate does not form, add sodium sulfite in approximately 0.1 g portions until a solid residue remains after repeated shaking.

11.8.3.4.4.Add 5 mL organic free reagent water and shake for 1 minute. Allow sample to stand for 5-10 minutes. (Centrifuge if necessary to separate the layers). Transfer the sample extract (top layer) to a vial. The final volume is defined as 1.0 mL in section 11.8.3.4.1.

11.8.4. Sulfuric Acid Cleanup

11.8.4.1.Add approximately 2-5 mL of concentrated sulfuric acid to 2 mL of sample extract in a Teflon capped vial.

Caution: There must be no water present in the extract or the reaction may shatter the sample container.

11.8.4.2.Shake or vortex for about thirty seconds and allow to settle. (Centrifuge if necessary)

11.8.4.3.Remove the sample extract (top layer) from the acid using a Pasteur pipet and transfer to a clean vial. ***CAUTION:*** It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.

11.8.4.4.If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

11.8.4.5.*Dispose of the sulfuric acid waste to the waste sulfuric acid drum for recycling.*

11.8.5. Acid/Base Partition Cleanup

11.8.5.1.Place 10 mL of the solvent extract from a prior extraction procedure into a 125 mL separatory funnel.

11.8.5.2.Add 20 mL of methylene chloride to the separatory funnel.

11.8.5.3.Slowly add 20 mL of DI water which has been previously adjusted to a pH of 12 to 13 with 10 N sodium hydroxide.

11.8.5.4.Seal and shake the separatory funnel for at least two minutes with periodic venting to release excess pressure.

CAUTION: Methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed.

11.8.5.5.Allow the organic layer to separate from the aqueous phase for a minimum of ten minutes.

11.8.5.6.If an emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.8.5.7.Separate the aqueous phase and transfer it to a 125 mL Erlenmeyer flask. Repeat the extraction two more times using

fresh 20 mL aliquots of dilute sodium hydroxide (pH 12-13). Combine the aqueous extracts.

11.8.5.8. Water-soluble organic acids and phenols will be primarily in the aqueous phase. Base/neutral analytes will be in the methylene chloride. If the analytes of interest are only in the aqueous phase discard the methylene chloride and proceed to Section 11.8.5.9. If the analytes of interest are only in the methylene chloride, discard the aqueous phase and proceed to Section 11.8.5.11.

11.8.5.9. Externally cool the flask with ice while adjusting the aqueous phase to a pH of 1-2 with sulfuric acid (1:1). Transfer the cool aqueous phase to a clean 125 mL separatory funnel.

11.8.5.10. Add 20 mL of methylene chloride to the separatory funnel and shake for at least two minutes. Allow the methylene chloride to separate from the aqueous phase and collect the methylene chloride in an Erlenmeyer flask. Repeat the extraction two more times using fresh methylene chloride and extracting at pH 1-2. Combine the three extracts.

11.8.5.11. Dry the extract by passing through a funnel containing 10-20 g anhydrous sodium sulfate. Rinse the funnel with an additional 20-30 mL of clean methylene chloride

11.8.5.12. Cover with aluminum foil if the extract is not concentrated immediately. Refer to section 11.7 for concentration.

11.8.5.13. Dispose of solvent and water remaining in the separatory funnel into the *LLE waste drum. Waste methylene chloride goes to the DCM drum for recycling.*

11.8.6. Silica Gel Cleanup

11.8.6.1. Cleanup of Extracts Prior to Determination of Polynuclear Aromatic Hydrocarbons by GC/FID.

11.8.6.1.1. Exchange the extract solvent to cyclohexane and reduce the volume to 2 mL prior to cleanup.

11.8.6.1.2. Prepare a slurry of approximately 10 g of activated silica gel in about 15 mL of methylene chloride. Place this into a 10 mm ID chromatographic column. Tap the column to

settle the silica gel. Add 1-2 cm of anhydrous sodium sulfate to the top of the silica gel.

11.8.6.1.3.Pre-elute the column with 40 mL of pentane. Elution rate should be approximately 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate to air, transfer the 2 mL cyclohexane sample extract onto the column using an additional 2 mL of cyclohexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 25 mL of pentane and continue the elution of the column. Discard this pentane eluate *to the flammable solvent waste drum.*

11.8.6.1.4.Elute the column with 25 mL of methylene chloride/pentane (2:3 v/v). Since the polynuclear aromatic hydrocarbons elute in this fraction, collect the eluate and concentrate to the required volume.

11.8.6.2.Cleanup of Extracts Prior to Determination of TPH Diesels by GC/FID.

11.8.6.2.1.Concentrate the extract to 5mLs (4mLs for soils) and split the extract in half, saving 2.5mLs (2.0mLs for soils) as an archived extract.

11.8.6.2.2.Prepare a slurry of approximately 10 g of activated silica gel in about 15 mL of methylene chloride. Place this into a 10 mm ID chromatographic column. Tap the column to settle the silica gel. Add 1-2 cm of anhydrous sodium sulfate to the top of the silica gel.

11.8.6.2.3.Pre-elute the column with 40 mL of methylene chloride. Elution rate should be approximately 2 mL/min. Discard the eluate *as waste methylene chloride for recycling* and just prior to exposure of the sodium sulfate to air, transfer the sample extract onto the column using an additional 2mL of methylene chloride to complete the transfer.

11.8.6.2.4.Elute the column with 60 mL of methylene chloride. Collect the eluate and concentrate to 2.5mLs (2.0mLs for soils).

11.8.6.3.Cleanup of Extracts Using a Silica Gel Cartridge Prior to Determination of TPH Diesels by GC/FID.

11.8.6.3.1.Transfer extracts to a 16 mL test tubes after KD concentration. Concentrate the extract to 2.0 mLs under nitrogen.

11.8.6.3.2.Set up the silica gel clean up manifold and attach silica gel cartridges.

11.8.6.3.3.Pre-elute the cartridge with 2 mL of methylene chloride. Elution rate should be approximately 2 mL/min, discard the eluate. Do not allow the cartridge to run dry. Place clean 16 mL test tubes into position in the manifold. Transfer the 2 mL extract onto the column using an additional 2 mL of methylene chloride to complete the transfer. Rinse the cartridge with an additional 2 mL of methylene chloride.

11.8.6.3.4.Collect the elutes and adjust to 3 mL under nitrogen. Samples are now ready for analysis.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method detection limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The procedure for the determination of the method detection limit is given in Quanterra QA Policy #: QA-005

13.2. Initial demonstration

Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The spiking level should be equivalent to a mid-level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. (CORP-GC-0001, CORP-MS-0001, 0002)

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

14.1. *Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.*

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated, and disposed according to the *waste streams detailed in the facility hazardous waste management procedures, Attachment B of the Chemical Hygiene Plan, Section WS002, Table 1, current edition.*

15.2. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment B of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis, current edition.

15.3. *Flammable* solvent waste must be disposed of *to the Flammable Liquid waste drum.*

15.4. *Sulfuric* acid waste must be collected in the *Waste Sulfuric Acid drum.*

15.5. Solid materials (soil, gloves, soiled paper products) are placed in the *laboratory trash* container. Do not put liquids in the solid waste container.

15.6. Methylene chloride saturated water and remaining sample after the extraction is *disposed* into *LLE waste drum.*

15.7. Waste mercury must be disposed of to the mercury labpack waste stream.

15.8. Expired standards must be rotated out of the laboratory for disposal to the Vial Waste stream.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, 3550B, 3600C, 3610B, 3620B, 3640A, 3650B, 3660B, AND 3665A.

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Aqueous sample volumes may be determined by weight.

17.1.3. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.1.4. 10 g of soil is used for pH determination, rather than the 50 g suggested in the reference method. The volume of water is also adjusted to maintain the sample / water ratio specified in the method.

17.2. Modifications from previous revision

17.2.1. Some details of separatory funnel extraction and continuous liquid-liquid extraction have been changed.

17.2.2. Recovery limits for GPC cleanup have been changed.

17.2.3. Solvent for organochlorine pesticide extractions has been changed to hexane / acetone.

17.2.4. A number of clarifications have been made

17.2.5. The appendix describing extraction of herbicides by method 8150 has been removed.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Tables

Table 1

Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270C	1-2	11-12
625	11-12	1-2
Pest/PCB: 8081A, 8082, 608	5-9	None
OPP: 8140	6-8	None
OPP: 8141	as received	None
Hydrocarbons: 8015	as received	None
PAH: 8310, 8100 & 610	as received	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a 4 replicate initial demonstration of capability and a method detection limit study. (See section 13). Client acceptance should be documented before using this option.

Table 2

Exchange Solvents and Final Volumes

Final Volumes and Exchange Solvents if no cleanup is used

Type	Exchange Solvent for Analysis	Final Volume for Analysis
Semivolatiles (8270C)	N/A	1.0
Pesticides (8081A, 8082)	Hexane	10.0
PAH by HPLC (8310)	Methanol	1.0 mL for aqueous; 2.0 mL for soil

PAH by GC (8100)	N/A	1.0
TPH (8015 Modified)	N/A	3.0
OPP (8140, 8141)	Toluene, hexane or isooctane	10.0

Final Volumes and exchange solvents if GPC cleanup is used			
Type	Exchange Solvent for GPC	Final Volume for GPC	Final Volume and solvent for Analysis
Semivolatiles (8270C)	CH_2Cl_2	10 mL[1]	0.5 mL CH_2Cl_2
Pesticides (8081A, 8082)	CH_2Cl_2	10 mL[1]	5 mL, hexane
OPP (8140, 8141)	CH_2Cl_2	10 mL	5 mL

[1] Final volume for GPC may be 4 mL if a 2 mL sample loop is used

Final volumes and exchange solvents if Florisil cleanup is used			
Type	Exchange Solvent for Florisil	Final Volume for Florisil	Final Volume and solvent for Analysis
Pesticides (8081A, 8082)	Hexane	10 mL (2 mL aliquot used)	4 mL, hexane

Final volumes and exchange solvents if both GPC and Florisil cleanup are used					
Type	Exchange Solvent for GPC	Final Volume for GPC	Exchange Solvent for Florisil	Final Volume for Florisil	Final volume for analysis

Pesticides (8081A, 8082)	Methylene Chloride	10 mL	Hexane	2.5 mL (2 mL aliquot used)	2 mL, hexane

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

Note: If silica gel cleanup is performed, exchange solvent to cyclohexane prior to cleanup.

Aliquot Volumes		
Type	Following KD Concentration	Prior to Florisil
Pesticides (8081A, 8082)	10 mL; aliquot 5 mL	2.5 mL; aliquot 2 mL
OPP (8140, 8141)	10 mL; aliquot 5 mL	NA
PAH by HPLC (Soils only, 8310)	3 mL; aliquot 1 mL	NA

Table 3			
Surrogate Spiking Solutions			
Analyte Group	Surrogate Spike Solution ID	Volume (mL) Water	Soil
BNA (8270C)	100/150 ppm BNA	0.5	0.5
Pest (8081A)	0.2 ppm DCB/TCX 3/90 P-Surro	1.0	2.0
Pest/PCB (8081A, 8082)	0.2 ppm DCB/TCX 3/90 P-Surro	1.0	2.0
PCB (8082)	0.2 ppm DCB/TCX 3/90 P-Surro	1.0	2.0
OPP (8140, 8141)	10 ppm Ethion	0.25	0.5
OPP (Appendix IX, 8141)	10 ppm Ethion	0.25	0.5
TPH Diesel (8015 Mod.)	400 ppm OTP	0.10	0.10
PAH by HPLC (8310)	500 ppm 1-Methylnaphthalene	0.05	0.20
BNA (AFCEE) (8270C)	100/200 ppm BNA	1.0	1.0

Table 4

Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL) Water	Volume (mL) Soils
BNA (8270C)	100/150 ppm BNA	0.5	0.5
BNA TCLP (1311/8270C)	BNA TCLP Spike	1.0	1.0
BNA NPDES (625)	BNA NPDES Spike	1.0	1.0
Pest (8081A, 8082)	0.5/1.0 ppm Pest 3/90 P-matrix	1.0	1.0
Pest TCLP (1311/8081A)	Pest 3/90 P-matrix	1.0	1.0
Pest NPDES (608)	Pest NPDES Spike	1.0	1.0
Pest/PCB (8081A/8082)	1.0 ppm pest spike 3/90 P-matrix	1.0	1.0
PCB (8082)	10 ppm PCB Spike Aroclor 1660	1.0	1.0
BNA (AFCEE) (8270C)	100/200 GAFB BNA Spike	1.0	1.0
Pest (AFCEE) (8081A)	0.4/1.0 ppm INDA/B Spike	0.5	2.0
OPP (8140,8141)	10 ppm 8140 Spike	0.25	0.5
OPP (Appendix IX, 8141)	5 ppm 8140 AP9 Spike	0.5	1.0
PAH by HPLC (8310)	2.0/100 ppm 8310 Spike	0.10	0.40
TPH Diesel (8015 Mod.)	3000 ppm Diesel Spike	0.10	0.10

Table 5

Surrogate Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
BNA (8270C)	2-Fluorobiphenyl	Methanol	100
	Nitrobenzene-d5		100
	p-Terphenyl-d14		100
	2-Fluorophenol		150
	Phenol-d6		150

Table 5

Surrogate Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	2,4,6-Tribromophenol		150
	1,2-Dichlorobenzene-d4		100
	2-Chlorophenol-d4		150
Pest/PCB (8081A/8082)	Decachlorobiphenyl	Acetone	0.2
	Tetrachloro-m-xylene		0.2
OPP (8141)	Ethion	Acetone	10
OPP (Appendix IX, 8141)	Ethion	Acetone	10
TPH Diesel (8015 Mod.)	OTP	Acetone	400
PAH by HPLC (8310)	1-Methylnaphthalene	Methanol	500

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
TCL BNA (8270C)	Acenaphthene	Methanol	100
	4-Chloro-3-Methylphenol		150
	2-Chlorophenol		150
	1,4-Dichlorobenzene		100
	2,4-Dinitrotoluene		100
	4-Nitrophenol		150

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	N-Nitroso-Di-n-Propylamine		100
	Pentachlorophenol		150
	Phenol		150
	Pyrene		100
	1,2,4-Trichlorobenzene		100
BNA TCLP (8270C)	1,4-Dichlorobenzene	Methanol	100
	2,4-Dinitrotoluene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	2-Methylphenol		100
	3-Methylphenol		100
	4-Methylphenol		100
	Nitrobenzene		100
	Pentachlorophenol		100
	Pyridine		100
	2,4,5-Trichlorophenol		100
	2,4,6-Trichlorophenol		100
BNA NPDES (625)		Methanol	
	Acenaphthene		100
	Acenaphthylene		100
	Anthracene		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Benzo(a)anthracene		100
	Benzo(b)fluoranthene		100
	Benzo(k)fluoranthene		100
	Benzo(a)pyrene		100
	Benzo(ghi)perylene		100
	Benzyl butyl phthalate		100
	Bis(2-chloroethyl)ether		100
	Bis(2-chloroethoxy)methane		100
	Bis(2-ethylhexyl)phthalate		100
	Bis(2-chloroisopropyl)ether		100
	4-Bromophenyl phenyl ether		100
	2-Chloronaphthalene		100
	4-Chlorophenyl phenyl ether		100
	Chrysene		100
	Dibenzo(a,h)anthracene		100
	Di-n-butylphthalate		100
	1,3-Dichlorobenzene		100
	1,2-Dichlorobenzene		100
	1,4-Dichlorobenzene		100
	3,3'-Dichlorobenzidine		100
	Diethyl phthalate		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Dimethyl phthalate		100
	2,4-Dinitrotoluene		100
	2,6-Dinitrotoluene		100
	Di-n-octylphthalate		100
	Fluoranthene		100
	Fluorene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	Indeno(1,2,3-cd)pyrene		100
	Isophorone		100
	Naphthalene		100
	Nitrobenzene		100
	N-Nitrosodi-n-propylamine		100
	Phenanthrene		100
	Pyrene		100
	1,2,4-Trichlorobenzene		100
	4-Chloro-3-methylphenol		100
	2-Chlorophenol		100
	2,4-Dichlorophenol		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	2,4-Dimethylphenol		100
	2,4-Dinitrophenol		100
	2-Methyl-4,6-dinitrophenol		100
	2-Nitrophenol		100
	4-Nitrophenol		100
	Pentachlorophenol		100
	Phenol		100
	2,4,6-Trichlorophenol		100
BNA (AFCEE, 8270C)		Methanol	
	N-Nitrosodimethylamine		100
	Phenol		200
	Bis (2-chloroethyl) Ether		100
	2-Chlorophenol		200
	1,3-Dichlorobenzene		100
	1,4-Dichlorobenzene		100
	Benzyl alcohol		100
	1,2-Dichlorobenzene		100
	2-Methylphenol		200
	2,2-Oxybis(1-chloropropane)		100
	4-Methylphenol		200
	N-Nitroso-di-n-propylamine		100
	Hexachloroethane		100
	Nitrobenzene		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Isophorone		100
	2-Nitrophenol		200
	2,4-Dimethylphenol		200
	Bis (2-chloroethoxy)methane		100
	2,4-Dichlorophenol		200
	Benzoic Acid		200
	1,2,4-Trichlorobenzene		100
	Naphthalene		100
	4-Chloroanaline		100
	Hexachlorobutadiene		100
	4-Chloro-3-Methylphenol		200
	2-Methylnaphthalene		100
	Hexachlorocyclopentadiene		100
	2,4,6-Trichlorophenol		200
	2,4,5-Trichlorophenol		200
	2-Chloronaphthalene		100
	2-Nitroanaline		100
	Dimethylphthalate		100
	Acenaphthylene		100
	2,6-Dinitrotoluene		100
	3-Nitroanaline		100
	Acenaphthene		100
	2,4-Dinitrophenol		200
	Dibenzofuran		100
	4-Nitrophenol		200
	2,4-Dinitrotoluene		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Diethylphthalate		100
	Fluorene		100
	4-Chlorophenyl-phenyl ether		100
	4-Nitroanaline		100
	4,6-Dinitro-2-methylphenol		200
	N-Nitrosodiphenylamine		100
	Azobenzene		100
	4-Bromophenyl-phenyl ether		100
	Hexachlorobenzene		100
	Pentachlorophenol		200
	Phenanthrene		100
	Anthracene		100
	Carbazole		100
	Di-n-Butylphthalate		100
	Fluoranthene		100
	Benzidine		100
	Pyrene		100
	Butylbenzylphthalatate		100
	Benzo (a) anthracene		100
	3,3-Dichlorobenzidene		100
	Chrysene		100
	Bis(2-ethylhexyl)phathalate		100
	Di-n-octyl phthalate		100
	Benzo(b)fluoranthene		100
	Benzo(k)fluoranthene		100
	Benzo(a)pyrene		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Indeno(1,2,3-cd)pyrene		100
	Dibenz(a,h)anthracene		100
	Benzo(g,h,i)perylene		100
Pest (8081A)	Aldrin	Acetone	0.5
	gamma-BHC (Lindane)		0.5
	4,4'-DDT		1.0
	Dieldrin		1.0
	Endrin		1.0
	Heptachlor		0.5
Pest TCLP (8081A)	Heptachlor	Acetone	0.5
	Heptachlor epoxide		0.5
	Lindane		0.5
	Endrin		0.5
	Methoxychlor		1.0
Pest NPDES (608)	Aldrin	Acetone	0.2
	alpha-BHC		0.2
	beta-BHC		0.2
	delta-BHC		0.2
	gamma-BHC (Lindane)		0.2
	4,4'-DDD		1.0
	4,4'-DDE		0.2
	4,4'-DDT		1.0

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Dieldrin		0.2
	alpha-Endosulfan		0.2
	beta-Endosulfan		1.0
	Endosulfan Sulfate		1.0
	Endrin		1.0
	Heptachlor		0.2
	Heptachlor Epoxide		0.2
PCB only (8082)	PCB 1016/1260	Acetone	2.0
Pesticides (AFCEE, 8081A)		Acetone	
	α-BHC		0.40
	γ-BHC		0.40
	β-BHC		0.40
	Heptachlor		0.40
	δ-BHC		0.40
	Aldrin		0.40
	Heptachlor Epoxide		0.40
	γ Chlordane		0.40
	α Chlordane		0.40
	Dieldrin		1.0
	Endrin		1.0
	Endo II		1.0
	4,4'-DDT		1.0
	Methoxychlor		1.0

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
OPP (8141)		Methanol	
	Thimet		10
	Diazinon		10
	Methyl Parathion		10
	Ethion		10
	Malathion		10
	Ethyl Parathion		10
OPP (AFCEE, 8141)		Methanol	
	Dichlorvos		10
	Phosdrin		10
	Demeton I		10
	Prophos		10
	Dibrom (Naled)		10
	Thimet (Phorate)		10
	Demeton II		10
	Demeton I&II		10
	Diazinon		10
	Disulfoton (Disyston)		10
	Methyl Parathion		10
	Ronnel		10
	Fenthion		10
	Chlorpyrifos		10
	Trichloronate		10

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Merphos		10
	Tokuthion		10
	Tetrachlorvinphos		10
OPP by App. IX (8141)		Methanol	
	O,O,O-TEPT		5.0
	Thionazin		5.0
	Sulfotepp		5.0
	Thimet		5.0
	Disyston		5.0
	Dimethoate		5.0
	M-Parathion		5.0
	Parathion		5.0
	Ethion		5.0
	Famphur		5.0
PAH by HPLC (8310)		Methanol	
	Naphthalene		50
	Fluorene		10
	Pyrene		10
	Indeno(1,2,3-cd)pyrene		5
	Benzo(a)pyrene		5
PAH (AFCEE, 8310)		Methanol	

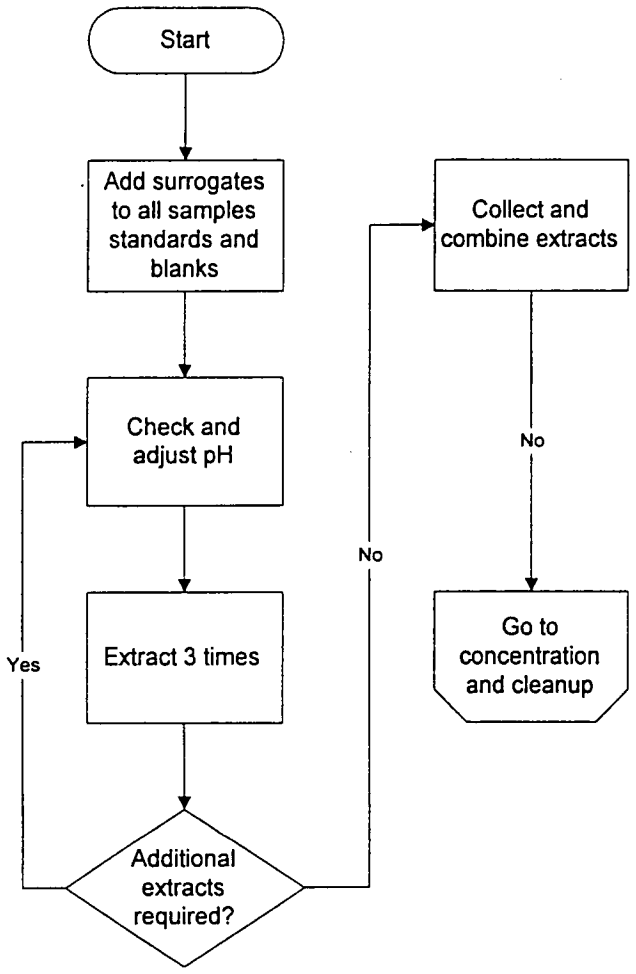
Table 6

Matrix Spike Components

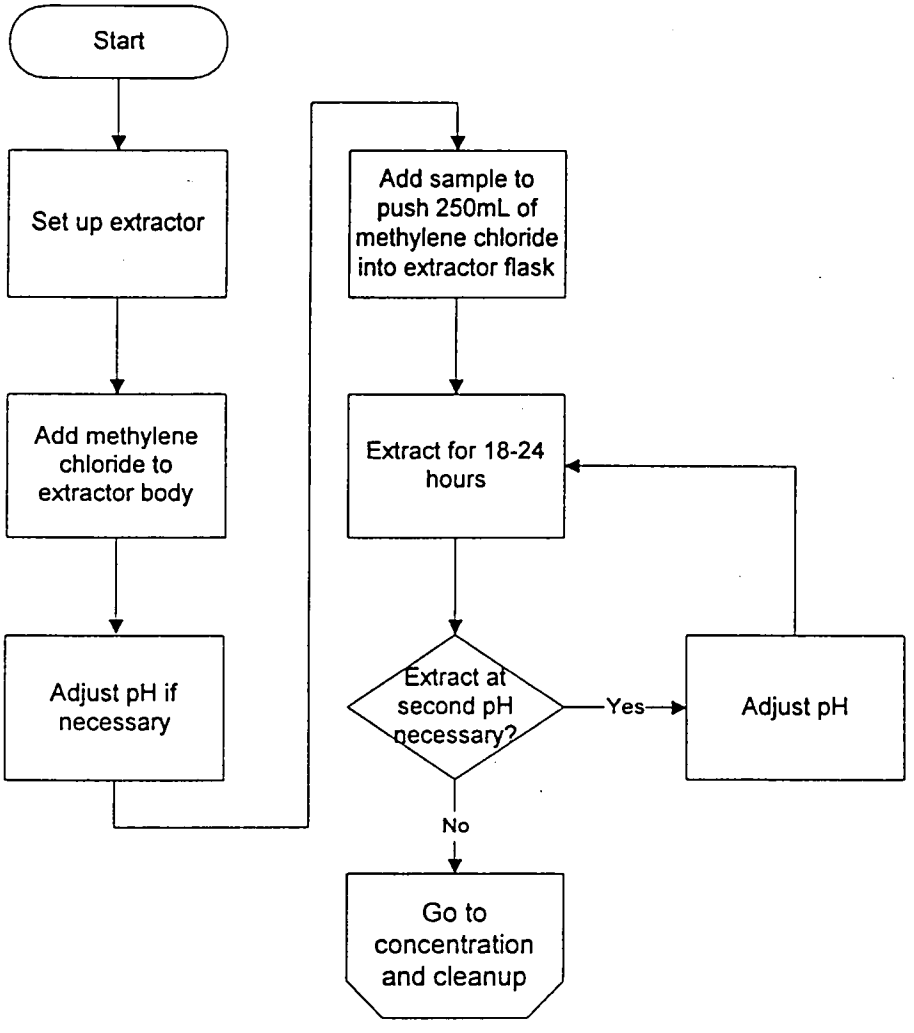
Type	Compounds	Solvent	Conc. (µg/mL)
	Naphthalene		50
	Acenaphthylene		50
	Acenaphthene		100
	Fluorene		10
	Phenanthrene		4.0
	Anthracene		2.0
	Fluoranthene		5.0
	Pyrene		10
	Benzo(a)anthracene		5.0
	Chrysene		5.0
	Benzo(k)fluoranthene		2.0
	Benzo(b)fluoranthene		2.0
	Dibenzo(a,h)anthracene		20
	Benzo(a,h,i)perylene		8.0
	Indeno(1,2,3-cd)pyrene		5.0
	Benzo(a)pyrene		5.0

17.3.1. Flow diagrams

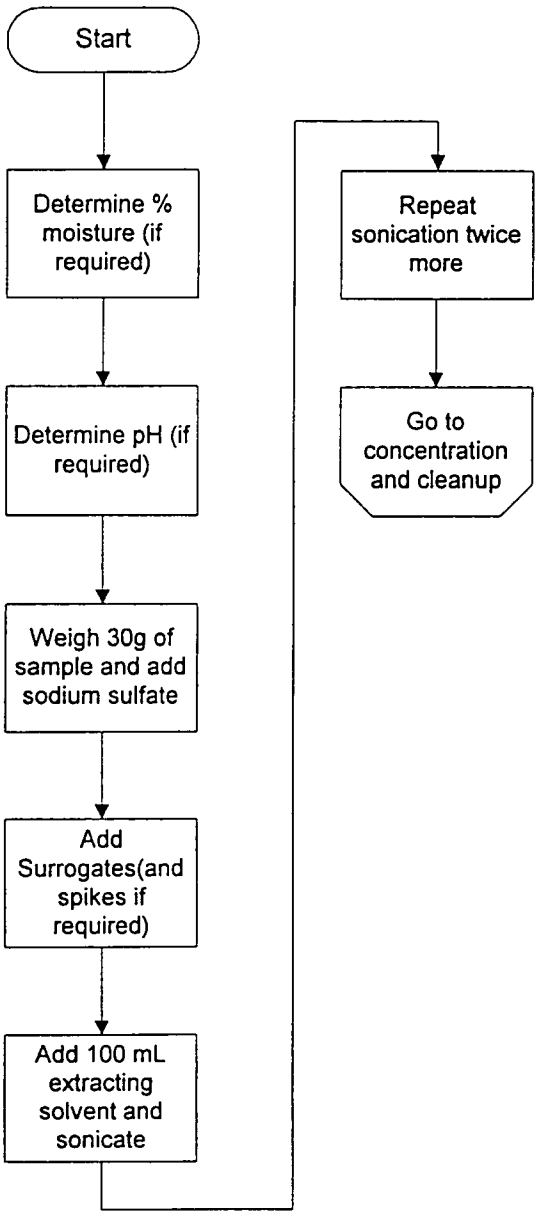
17.3.1.1.Separatory funnel extraction



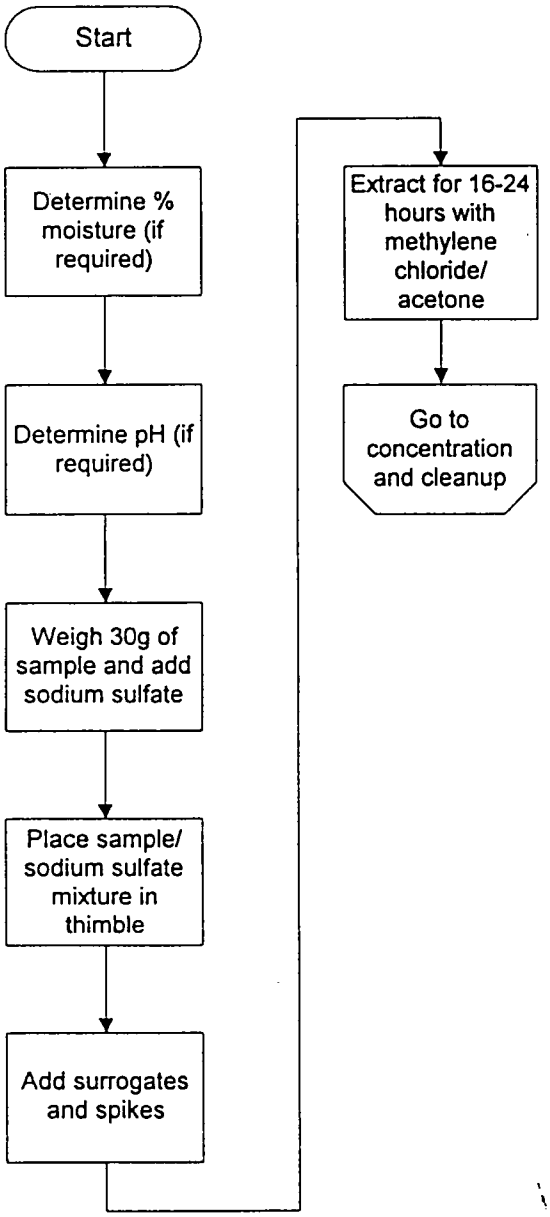
17.3.2. Continuous liquid/liquid extraction



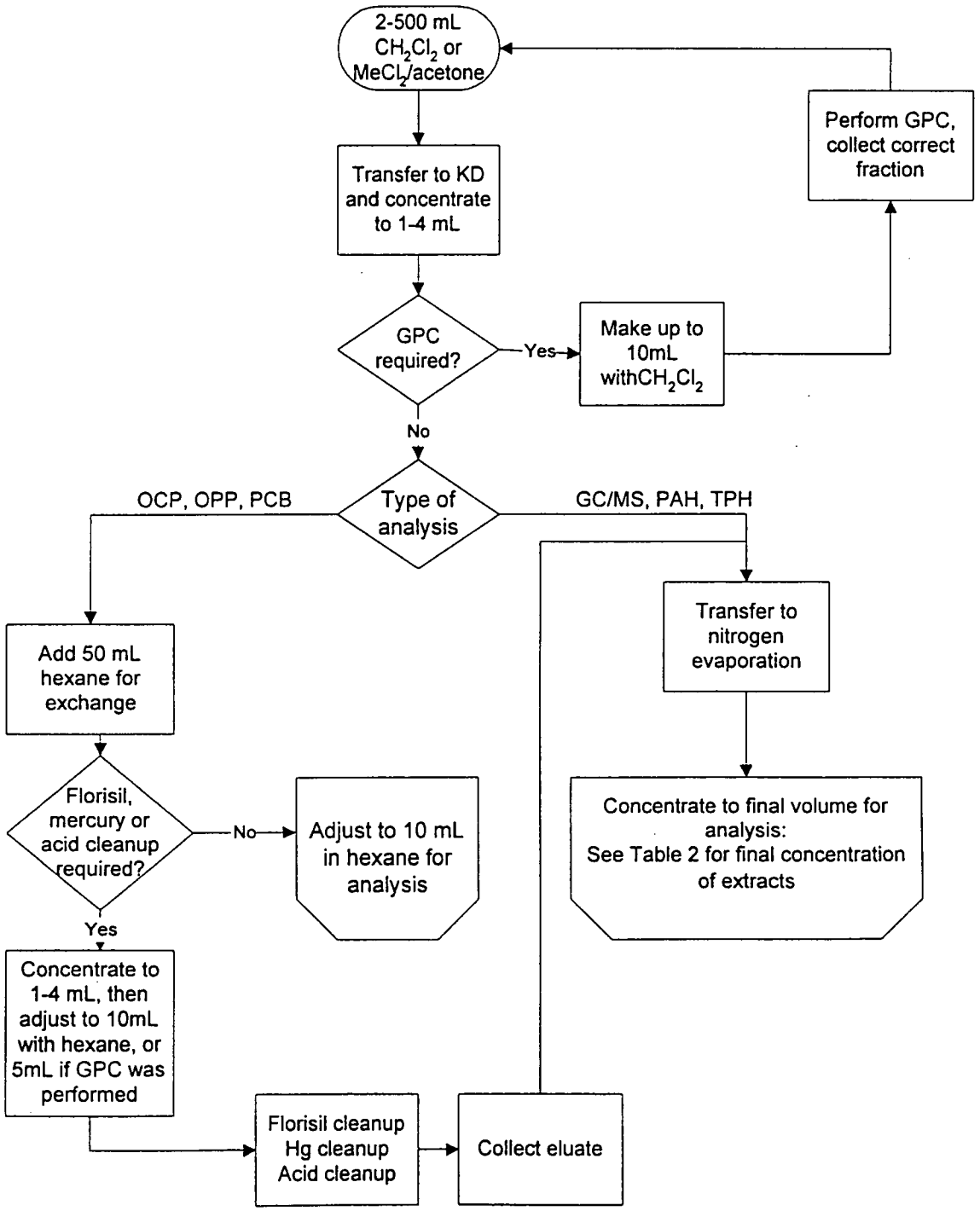
17.3.3. Sonication Extraction



17.3.4. Soxhlet extraction



17.3.5. Concentration and cleanup



1. SCOPE AND APPLICATION

This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and TCLP extracts. Appropriate compounds for extraction by this method are listed in CORP-GC-0001, Appendix D, Gas Chromatography of Phenoxy Acid Herbicides based on Method 8151A.

2. SUMMARY OF METHOD

This method is based on SW846 method 8151A. Aqueous samples are hydrolyzed if esters and acids are to be determined, then washed with methylene chloride by a separatory funnel extraction. After acidifying the sample the free acids are extracted into diethyl ether. Solids are extracted into methylene chloride/ acetone by sonication. If esters and acids are to be determined, the extract is hydrolyzed and extracted into diethyl ether. For both soils and aqueous samples, the free acid herbicides in the ether extract are esterified. The final volume is adjusted to prepare the extract for gas chromatography.

3. DEFINITIONS

Refer to section 3 of the main body of this SOP.

4. INTERFERENCES

Refer to section 4 of the main body of this SOP.

5. SAFETY

5.1. Refer to section 5 of the main body this SOP for basic safety information.

5.2. **DIAZOMETHANE is an extremely toxic gas with an explosion potential.** Since the explosion potential is catalyzed by imperfections in glass, generation of diazomethane must be carried out in glassware free of scratches, cracks, chips and which does not have ground glass joints. Solutions of diazomethane will be kept at temperatures below 90°C. Diazomethane must be generated and handled in a fume hood.

5.3. Diethyl ether is extremely flammable. It also tends to form peroxides when exposed to air. The peroxides can present an explosion hazard, especially when the ether is concentrated.

5.4. Diethyl ether must be free of peroxides as demonstrated by EM (or equivalent) Quant test strips. This test can be done every time the ether is used or once per week if the bottle is marked with the test date(s).

5.4.1. *To alert the user of potential peroxides that have formed in aging diethyl ether, date stamp the when opened.*

5.5. Concentrated potassium hydroxide solution is highly caustic.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the main body of this SOP for basic extraction equipment and supplies. Additional equipment and supplies needed for this procedure are listed below.

6.2. Diazomethane generation apparatus

6.3. EM Peroxide test strips

7. REAGENTS AND STANDARDS

7.1. Reagents are listed in Section 7 of the main body of this SOP. Additional Reagents and standards needed for this procedure are listed below.

7.2. Reagents

7.2.1. Potassium hydroxide solution, 37% aqueous solution, (w/v): Dissolve 37 g of potassium hydroxide pellets in reagent water and dilute to 100 mL. ***Caution:*** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

7.2.2. Sodium hydroxide solution, 6N. Dissolve 400 g NaOH in reagent water and dilute to 1.0L. ***Caution:*** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

7.2.3. Sodium hydroxide solution, 0.1N. Dissolve 4g NaOH in reagent water and dilute to 1.0L. Other volumes of solution may be made up as convenient.

7.2.4. Sulfuric acid, 1:1 Slowly add 500 mL concentrated sulfuric acid to 500 mL water. ***Caution:*** Considerable heat will be generated. The acid must be added to the water. Wear protective clothing and safety glasses. Other volumes of solution may be made up as convenient.

Note: Use a face shield and safety glasses when handling bulk corrosives and solvents.

7.2.5. Diazald, 99% purity

7.2.6. 2,3,4,5,6-Pentafluorobenzyl bromide (PFBBr)$C_6H_5CH_2Br$

7.2.7. Sodium sulfate, Na_2SO_4, Anhydrous, granular, acidified: Heat sodium sulfate in a shallow tray at 400°C for a minimum of 4 hours to remove phthalates and other interfering organic substances. In a large beaker, acidify by slurrying 1000 g sodium sulfate with just enough diethyl ether to cover. Add 2-5 mL of concentrated sulfuric acid and mix thoroughly. Place the mixture on a steam bath in a hood to evaporate the ether, or allow the ether to evaporate overnight. Larger or smaller batches of acidified sodium sulfate may be prepared using the reagents in the same proportions.

7.2.8. Sodium Chloride, NaCl

7.2.9. Acidified 5% sodium sulfate solution

Add 50 g of sodium sulfate to one liter of reagent water. Add 10 mL of concentrated H_2SO_4. (This reagent may be prepared in different quantities if the proportions are kept the same).

7.2.10. Diethyl ether, reagent grade.

7.2.11. Methanol, reagent grade.

7.2.12. Silicic acid

7.3. Standards

7.3.1. Surrogate Standard

See Table A3.

7.3.2. Matrix Spike and LCS standard

See Table A4.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Sample collection and storage is described in Section 8 of the main body of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the main body of this SOP for Quality control procedures.

10. CALIBRATION AND STANDARDIZATION

Not applicable

11. PROCEDURE

11.1. Preparation of Aqueous Samples

11.1.1. Weigh the sample bottle and pour approximately 1 liter (100 mL for TCLP leachates) into a 2 liter separatory funnel. Reweigh the bottle and record the sample volume on the benchsheet, assuming a density of 1.0. Alternatively, measure 1 liter in a graduated cylinder. If less than 1 liter was used, add reagent water to make the volume up to 1 liter.

11.1.2. Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1.0 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

11.1.3. Add 250 g of NaCl to the sample and shake to dissolve the salt.

11.1.4. Hydrolysis

Use this step only if herbicide esters in addition to herbicide acids are to be determined. This is normally the case. If the herbicide esters are not to be determined, omit this step and go to 11.1.10.

11.1.4.1.Add 17 mL of 6N NaOH to the sample, seal and shake. Check the pH of the sample with pH paper. If the pH of the sample is not $\geq$12 adjust to $\geq$12 by adding more NaOH. Let the sample sit at room temperature for 2 hours to complete the hydrolysis.

11.1.5. If the sample was originally in a 1 liter bottle, and the whole sample was used, add 60 mL of methylene chloride to the sample bottle. Rinse the bottle (or graduated cylinder, if used) and add the methylene chloride to the separatory funnel.

11.1.6. If the whole contents of the sample bottle were not used, add 60 mL of methylene chloride to the separatory funnel.

11.1.7. Extract the sample by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used). Allow the organic layer to separate from the aqueous layer. If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

11.1.8. Discard the **methylene chloride** phase. *Dispose the waste methylene chloride to the DCM drum.*

11.1.9. Add a second 60 mL of methylene chloride and repeat the extraction a second time, discarding the methylene chloride. Repeat the extraction a third time.

11.1.10. Add 17 mL of cold (4°C) 1:1 sulfuric acid to the sample. Seal, and shake to mix. Check the pH of the sample with pH paper. If the pH is not ≤ 2, and more acid to adjust the pH to ≤ 2.

Caution: Addition of acid may cause heat and / or pressure build up.

11.1.11. Add 120 mL diethyl ether to the sample and extract by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used). Allow the organic layer to separate from the aqueous layer. If a emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

11.1.12. Drain the aqueous layer into a clean flask or beaker. Collect the ether phase in a clean flask or bottle containing approximately 10g of acidified anhydrous sodium sulfate.

11.1.13. Return the aqueous phase to the separatory funnel, add 60 mL diethyl ether and repeat the extraction procedure a second time., combining the ether extracts. Repeat the extraction a third time with 60 mL diethyl ether. Discard the aqueous phase after the third extraction.

11.1.14. Allow the extract to remain in contact with the acidified sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the acidified sodium sulfate solidifies in a cake, add additional acidified sodium sulfate. The amount of acidified sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken.

11.1.15. Proceed to section 11.5, concentration.

11.2. Extraction of soil and sediment samples

11.2.1. Decant and discard any water layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator). Document if a water layer was discarded.

11.2.2. Weigh 50.0 g of moist solid sample into an clean glass jar. Use 50 g of sodium sulfate for the Method Blank and 50g Ottawa sand for the LCS. Acidify the sample with 5 mL of concentrated HCl.

11.2.3. There should be a small amount of liquid phase. If not, add reagent water until there is. Stir well with a spatula. (*Note:* This is not necessary for the method blank or LCS)

11.2.4. After 15 minutes, stir with a spatula and check the pH of the liquid phase. Add more acid if necessary to bring the pH to <2, repeating the stirring and standing time after each acid addition. (*Note:* The pH of the method blank and LCS are not determined.)

11.2.5. Add 60 g of acidified sodium sulfate and mix well. The sample should be free flowing. If not, add more acidified sodium sulfate.

11.2.6. Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1.0 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

11.2.7. Add a minimum of 100 mL of 1:1 methylene chloride:acetone to the beaker.

11.2.8. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.2.9. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: Hearing protection is required when sonicating.

11.2.10. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of acidified sodium sulfate to the funnel cup.

11.2.11. Place the prepared funnel on a collection apparatus. If the herbicide esters are *not* to be determined, the collection apparatus is a bottle or flask containing approximately 10 g of acidified sodium sulfate. If the herbicide esters *are* to be determined, (normally the case) the collection apparatus is glassware suitable for the hydrolysis step, typically a KD flask or Turbovap tube.

11.2.12. Decant and filter extracts through the prepared funnel into the collection apparatus.

11.2.13. Repeat the extraction two more times with additional 100 mL minimum portions of methylene chloride / acetone each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

Note: Alternatively, the three extracts may be collected together and then filtered through the acidified sodium

sulfate.

11.2.14. If the herbicide esters are not to be determined, dry the extract as described in 11.4.2 or go to cleanup, section 11.3. If the herbicide esters are to be determined (normally the case) proceed to section 11.2.15.

11.2.15. Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of water to the extract. Check the pH with pH paper. If the pH is not $\geq$12, adjust with additional KOH.

11.2.16. Heat on a water bath at 60-65°C for 2 hours. Allow to cool.

11.2.17. Transfer the solution to a separatory funnel and extract three times with 100 mL portions of methylene chloride. **Discard the methylene chloride phase** *to the waste methylene chloride drum.* The aqueous solution contains the herbicides.

11.2.18. Adjust the pH of the solution to $\leq$2 with 1:1 sulfuric acid.

11.2.19. Extract once with 40 mL diethyl ether and twice with 20 mL diethyl ether.

11.2.20. Proceed to section 11.3, Cleanup, if required, or Section 11.4, Extract drying.

11.3. Cleanup

This cleanup step may be necessary if the procedure for determining the herbicide acids only is being followed. It is not normally required if the acids and esters are being determined. (The usual case.) If cleanup is not required, proceed to section 11.4., Extract drying.

11.3.1. Prepare 45 mL of basic extraction fluid by mixing 30 mL of reagent water with 15 mL of 37% KOH. Use three 15 mL portions of this fluid to partition the extract from sectionusing a small separatory funnel. **Discard the organic phase.**

11.3.2. Adjust the pH of the solution to $\leq$2 with cold (4°C) sulfuric acid. (1:1). Extract once with 40 mL diethyl ether and twice with 20 mL diethyl ether.

Caution: Addition of acid may cause heat and / or pressure build up.

11.4. Extract drying

11.4.1. Combine the extracts and pour through a funnel containing acidified sodium sulfate into a flask or bottle containing approximately 10 g acidified sodium sulfate. Rinse the funnel with a little extra diethyl ether.

11.4.2. Allow the extract to remain in contact with the acidified sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the acidified sodium sulfate solidifies in a cake, add additional acidified sodium sulfate. The amount of acidified sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken. Proceed to section 11.5, concentration.

11.5. Concentration

11.5.1. Transfer the ether extract by decanting, or through a funnel plugged with acid washed glass wool and a small amount of acidified sodium sulfate, into a Turbovap concentrator tube or a 500 mL K-D flask equipped with a 10 mL concentrator tube. Use a stirring rod to crush the caked sodium sulfate during transfer. Rinse the flask or bottle with 20-30 mL ether to complete transfer.

11.5.2. Attach a three ball Snyder column to the K-D apparatus, prewet the column with a few mL of ether from the top, and place the apparatus on a water bath at approximately 60°C. At the proper rate of distillation, the balls of the column will chatter, but the chambers will not flood. When the apparent volume reaches 2 mL, remove from the water bath and allow to completely cool.

11.5.3. Transfer extracts to 16 mL test tubes. Nitrogen blow down the samples to between 0.5 mL and 1.0 mL

11.5.4. Carefully disassemble the concentrator tube and rinse the lower glass joint with a small amount of diethyl ether.

11.5.5. Add 0.1 mL of methanol.

11.5.6. The extract is now ready for esterification by either the diazomethane solution method (11.6) or the bubbler method (11.7)

11.6. Esterification (diazomethane solution method)

11.6.1. Preparation of Diazomethane solution

CAUTION: **Diazomethane is potentially explosive.**

- **A temporary shield or the sash of the hood must protect the face and body of the analyst.**
- **Never heat solutions of diazomethane above 90°C, due to the explosive potential.**
- **Do not use glass stirring rods or any glassware with ground glass joints, as this can initiate violent reaction or explosion.**
- **All glassware must be scrupulously cleaned and free from scratches, to avoid potential initiation of violent reaction or explosion.**

11.6.1.1. Weigh out 10.0 g potassium hydroxide in a 125 mL beaker. Add 16 mL water and 20 mL ethanol. Mix well until the potassium hydroxide is dissolved and pour into the reaction vessel.

11.6.1.2. Attach a 100 mL receiving flask to the condenser and cool the receiver in an ice bath.

11.6.1.3. Fill the condenser with dry ice, then add acetone slowly until the cold finger is about one third full.

11.6.1.4. Mix 10.0 g diazald and 90 mL ether. Place in a separatory funnel over the reaction vessel.

11.6.1.5. Warm the reaction vessel on the water bath to 50-60°C or until the ether begins to reflux. *CAUTION:* Do not allow the temperature to exceed 70°C. Add diazald solution over a period of 40 minutes. The rate of distillation should approximate the rate of addition. Replenish the cold finger

with dry ice as necessary.

11.6.1.6. When the diazald solution has been used up, slowly add 10 mL of ether and continue distillation until the distillate is colorless.

11.6.1.7. The diazomethane solution is documented with a lot number. It is stored in a freezer at -10 to -20°C and has an expiration date of six months.

11.6.2. Disposal of outdated diazomethane solutions

11.6.2.1. Add 20 mL of ether to 2 mL of glacial acetic acid in a large beaker in the hood.

11.6.2.2. Add the diazomethane solution slowly to the acetic acid. The yellow color of the diazomethane disappears as it reacts with the acetic acid. If the yellow color persists at any time during the addition, and does not disappear with gentle swirling of the beaker, add additional acetic acid in ether before continuing the diazomethane addition.

11.6.2.3. Dispose of the ether solution in the non-chlorinated wastes bottle.

11.6.3. Esterification (Diazomethane solution method)

11.6.3.1. Add approximately 1 mL of diazomethane solution and let sit for 10-15 minutes.

11.6.3.2. Add approximately 0.2 g of silicic acid to the extract. Allow to spontaneously evaporate to about 1.0 mL, then make up to 12 mL with hexane. Nitrogen blow the sample down to a final volume of 10 mL.

11.6.3.3. Extract is ready for analysis by gas chromatography.

11.7. Esterification (Bubbler Method)

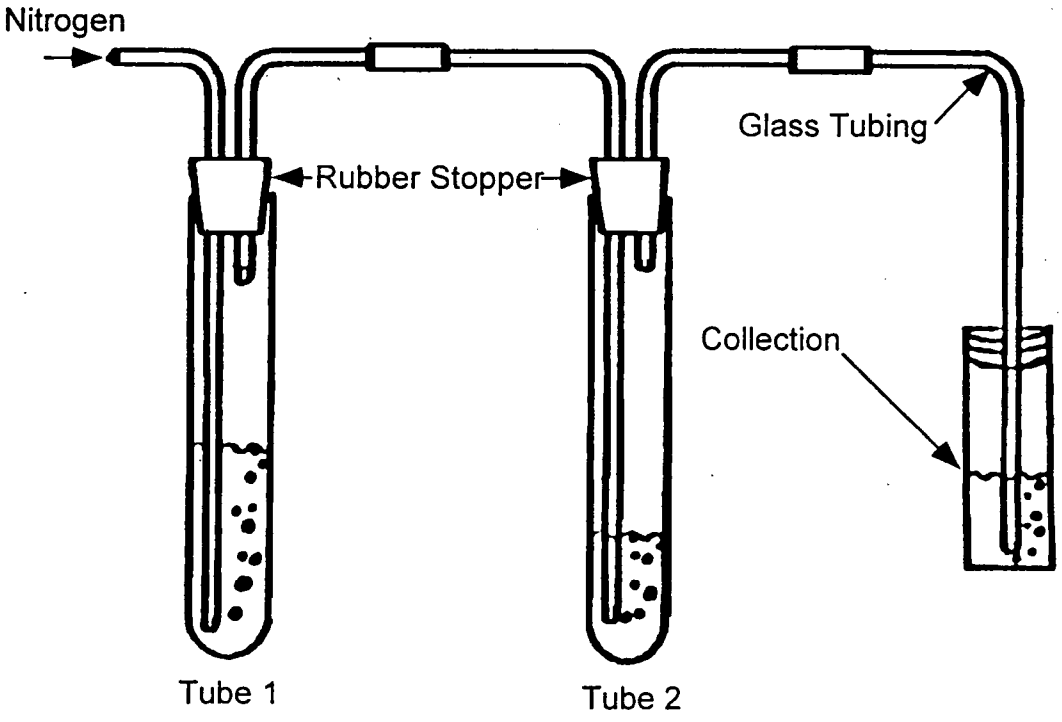
11.7.1. Assemble the diazomethane apparatus (see figure below) in a hood. Add 10 mL of diethyl ether to tube 1. Add 5 mL of 2% methanolic

KOH, 3 mL of ether and 0.5-1 g of diazald to tube 2.

11.7.2. Place the tip of the disposable pipette into the vial containing the first sample extract. Apply nitrogen flow (approx. 10 mL/min) to bubble diazomethane through the sample extract for about 1 minute, or until the yellow color persists. Replace the disposable pipette and place the tip into the vial containing the second extract. Continue until the diazald is consumed. (An additional 0.1-0.5 g diazald may be added to extend the generation of diazomethane).

11.7.3. Allow the extracts to stand for 20 minutes, then add approximately 0.2 g of silicic acid to each extract. Allow to stand for an additional 20 minutes.

11.7.4. Adjust the volume to 10 mL with hexane. The sample is now ready for gas chromatography.



12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

Refer to CORP-GC-0001 for details of method performance.

14. POLLUTION PREVENTION

Refer to section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

Refer to section 15 of the main body of this SOP.

16. REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Chlorinated Herbicides, Method 8151A.

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Directions to add sufficient reagent water to the soil sample so that the pH can be measured have been added (Section 11.1.2)

17.1.2. The bubbler esterification method uses methanolic KOH in place of the aqueous KOH / carbitol mixture recommended in method 8150B. This has been found to provide a more effective and reliable esterification.

17.2. Modifications from previous revisions

17.2.1. References have been updated

17.3. Tables

Table A1 Herbicide Surrogate Spiking Solutions		
Analyte Group	Surrogate Spike Solution ID	Volume (mL)
Herbicides	Herbicides SS	1.0

Table A2 Herbicide Matrix Spike and LCS Solutions		
Analyte Group	Matrix Spike Solution ID	Volume (mL)
Herbicides	Herbicides MS	1.0

Table A3 Herbicide Surrogate Spike Components			
Type	Compounds[1]	Solvent	Conc. (ug/mL)
Herbicides SS	2,4-DCAA	Methanol	16

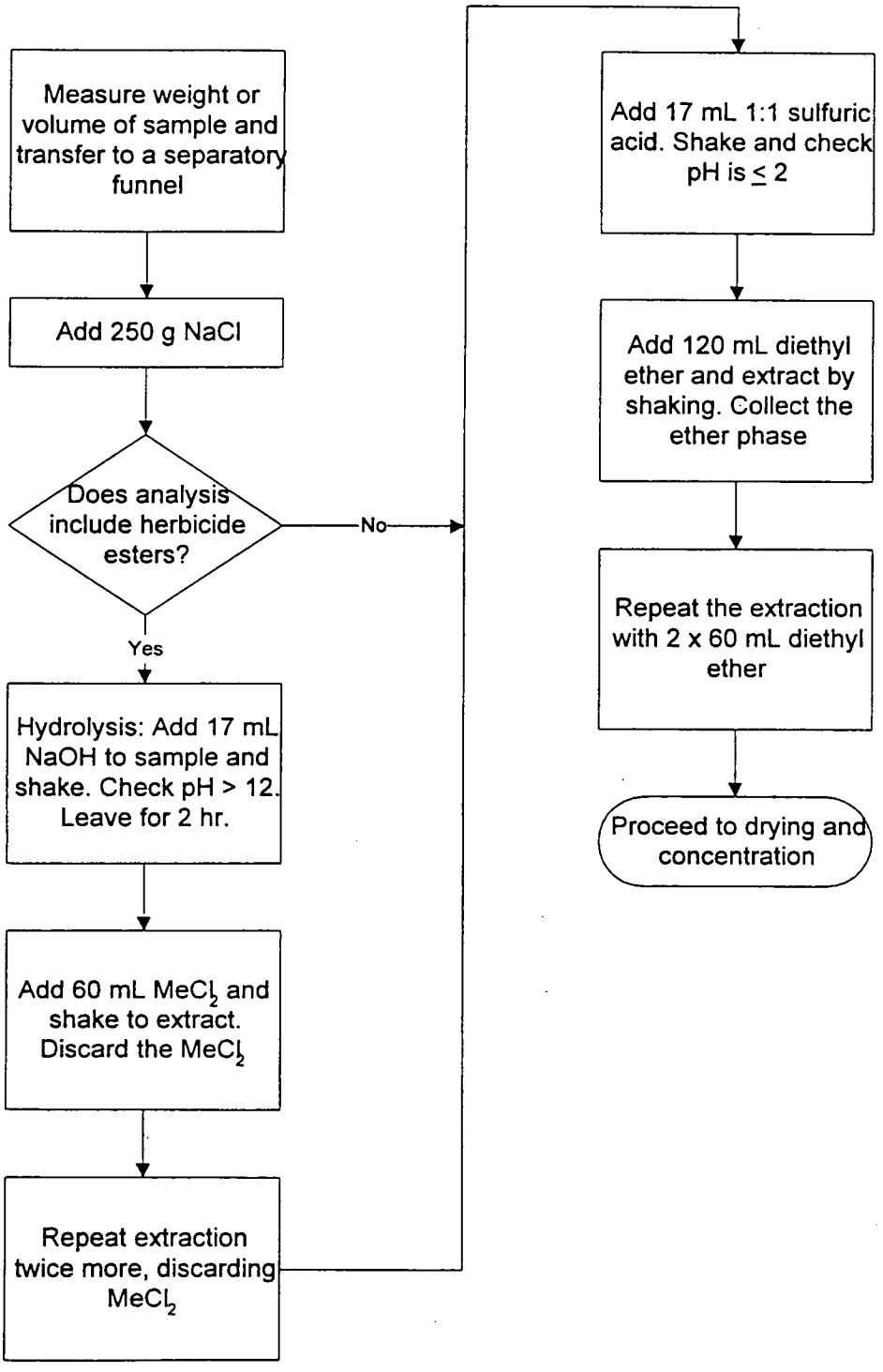
[1]The surrogate is spiked as the free acid

Table A4 Herbicide Matrix Spike Components			
Type	Compounds[1]	Solvent	Conc. (ug/mL)
Herbicides MS	2,4-D	Methanol	16
	2,4-DB		16
	2,4,5-TP (Silvex)		4

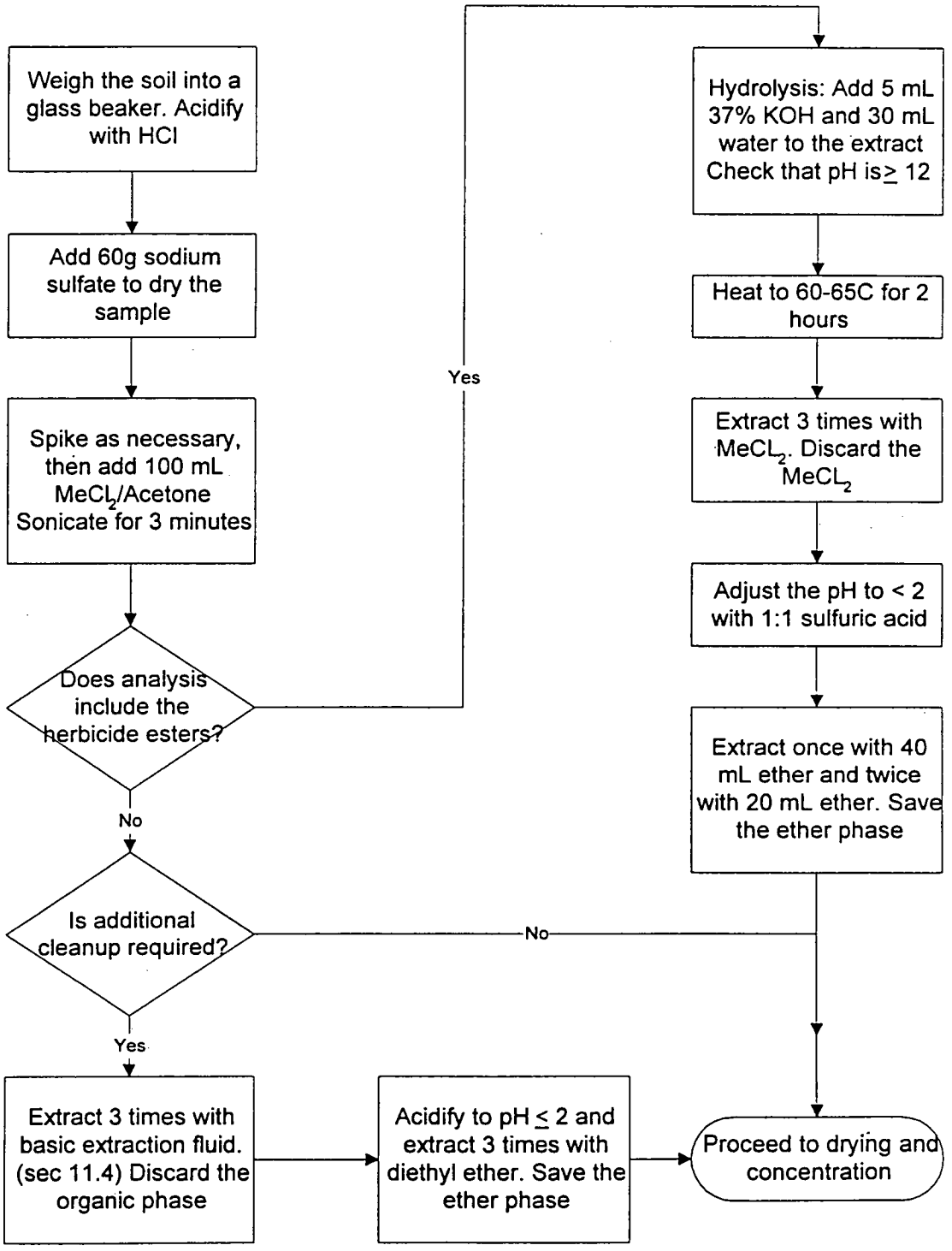
Table A4			
Herbicide Matrix Spike Components			
Type	Compounds[1]	Solvent	Conc. (ug/mL)
	Dalapon		8
	2,4,5-T		4

[1]The herbicide spiking solution contains the herbicides as the free acids.

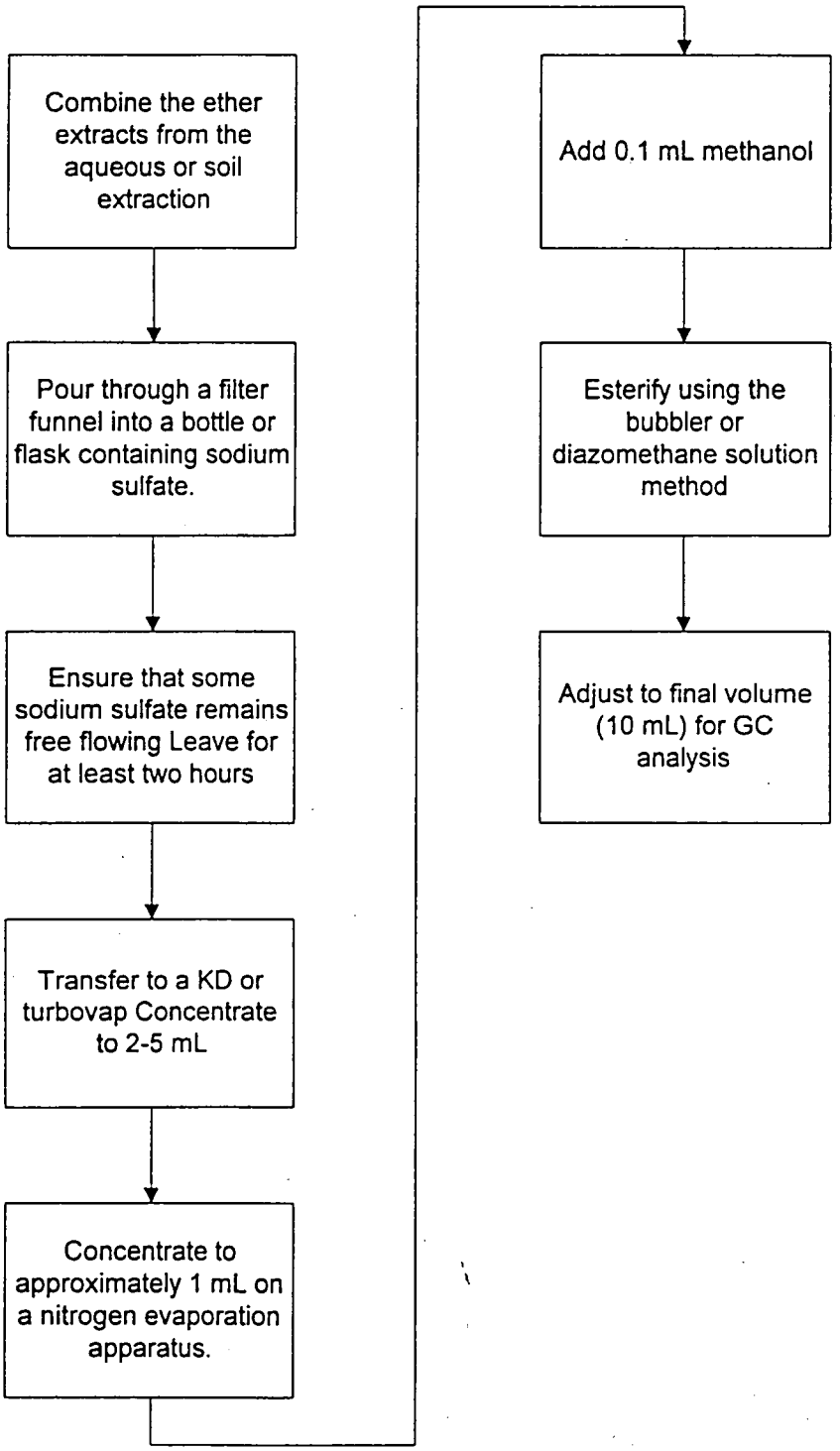
Extraction of Aqueous Samples



Extraction of Soils and Sediments


Weigh the soil into a glass beaker. Acidify with HCl
Add 60g sodium sulfate to dry the sample
Spike as necessary, then add 100 mL MeCL2/Acetone Sonicate for 3 minutes
Does analysis include the herbicide esters?
Yes
No
Is additional cleanup required?
No
Yes
Extract 3 times with basic extraction fluid. (sec 11.4) Discard the organic phase
Acidify to pH ≤ 2 and extract 3 times with diethyl ether. Save the ether phase
Hydrolysis: Add 5 mL 37% KOH and 30 mL water to the extract Check that pH is≥ 12
Heat to 60-65C for 2 hours
Extract 3 times with MeCL2. Discard the MeCL2
Adjust the pH to < 2 with 1:1 sulfuric acid
Extract once with 40 mL ether and twice with 20 mL ether. Save the ether phase
Proceed to drying and concentration

Drying, Concentration and Esterification



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Environmental Services

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QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B, 8021B, 8081A, 8082 and 8151A, SW-846

(SUPERSEDES: Revision 4)

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TABLE OF CONTENTS

1.	Scope and Application	4
2.	Summary of Method	4
3.	Definitions	4
4.	Interferences	4
5.	Safety	4
6.	Equipment and Supplies	4
7.	Reagents and Standards	5
8.	Sample Preservation and Storage	5
9.	Quality Control	5
10.	Calibration and Standardization	8
11.	Procedure	14
12.	Data Analysis and Calculations	16
13.	Method Performance	19
14.	Pollution Prevention	20
15.	Waste Management	20
16.	References	20
17.	Miscellaneous	20

List of Appendices:

Appendix A Analysis of Volatile Organics based on Method 8021B

Appendix B Analysis of Organochlorine Pesticides based on Method 8081A

Appendix C Analysis of Organochlorine Pesticides and PCBs based on Method 8082

Appendix D Analysis of Phenoxy Acid Herbicides based on Method 8151A

List of Tables

Table A1 Standard analyte lists for 8021B

Table A2-A4 Recommended conditions for method 8021B

Table A5 Surrogate and Internal standard concentrations for aqueous and low level soil samples, method 8021B

Table A6 Concentrations for LCS and MS/MSD compounds, low level soil and aqueous, method 8021B.

Table B1 Standard analyte and reporting limits, method 8081A

Table B2 Recommended conditions, method 8081A

Table B3 Calibration levels, method 8081A

Table B4 Column degradation evaluation mix, method 8081A

Table B5 LCS/matrix spike and surrogate levels, method 8081A

Table B6 LCS/matrix spike and surrogate levels for TCLP, method 8081A

Table B7 Suggested analytical sequence, method 8081A

Table B8 Performance limits, method 8081A

Table C1 Standard analyte list, method 8082

Table C2 Instrumental conditions, method 8082

Table C3 Calibration standards, method 8082

Table C4 LCS/Matrix spike and surrogate levels, method 8082

Table C5 Suggested Analytical Sequence, Method 8082

Table D1 Standard Analyte List, method 8151A

Table D2 Instrumental conditions, method 8151A

Table D3 Calibration standards, method 8151A

Table D4 LCS/Matrix spike and surrogate levels, method 8151B

Table D5 Performance limits, method 8151B

1. 1.SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices.

2. SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP # CORP-OP-0001) Solid samples are prepared using sonication, Soxhlet or pressurized fluid extraction (SOP # CORP-OP-0001). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. In addition, some purge and trap autosamplers are susceptible to port specific contamination. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates. The following requirements must be met:

Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded. Refer to the Quanterra Chemical Hygiene plan for a complete description of personal protection equipment.

5.1.1. N-Dex nitrile gloves provide varying degrees of intermittant splash protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. Specific hazards are covered in the appendices.

5.1.2. Standards must be handled in the Standards Preparation Hood.

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored at ≤6°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at t every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration.). Other volatile stock standards must be replaced every 6 months or sooner if comparison with check standards prepared from an independent source indicates a problem.

Standards must be rotated out of the lab to the Hazardous Waste storage area.

7.1.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If a vendor supplied standard has an earlier expiration date then that date is used.

7.1.2. All expired standards must be rotated out of the lab to the Hazardous Waste storage area.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at ≤6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Argon/Methane.

7.4. Quality control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

Semivolatile extracts must be refrigerated at ≤6°C and analyzed within 40 days of the end of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

9.2.1. Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted. In the event that multiple MS/SD are run within a batch due to client requirements, the additional MS/SD do not count toward the maximum 20 samples in a batch.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least semi-annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy QA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary is there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.4.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.4.3. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatiles samples, and sodium sulfate for semivolatiles soils tests (Refer to SOP No. CORP-OP-0001 for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of methanol as described in Appendix A. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.5.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.6. Instrument Blanks

9.6.1. An instrument blank must be analyzed during any 12 hour period of analysis that does not contain a method blank.

9.6.2. An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.6.3. Control criteria are the same as for the method blank, except that only reanalysis of affected samples would be required, not re-extraction.

9.7. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch; however, if the matrix spike and matrix spike duplicate are within limits, the batch may be acceptable.

9.7.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective action.

9.7.2. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.7.3. LCS compound lists are included in the appendices.

9.7.4. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.9. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or FID jets or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.2. With the exception of 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$Calibration\ Factor\ (CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\ (ng)}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this

approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_S = Response for the analyte to be measured
A_{iS} = Response for the internal standard
C_{iS} = Concentration of internal standard
C_S = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is $\leq$ 20%. The average %RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes.

10.6.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.6.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is $\leq$ 20%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.6.3. Linear regression

The linear fit uses the following functions:

10.6.3.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.6.3.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.6.4. Quadratic curve

The quadratic curve uses the following functions:

10.6.4.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.6.4.2. Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard error (%RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows:

$$\% RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

Note that when average response factors are used, %RSE is equivalent to %RSD.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be $\leq$ 20%.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r^2) and Coefficient of Determination (r) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have a very good correlation coefficient (>0.995), while also having > 100% error at the low point. (Edgerley, "Improving Calibration Accuracy in the Environmental Laboratory", in press).

10.9. Weighting of data points

10.9.1. In a quadratic calibration fit, the points at the lower end of the calibration curve have less relative variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the

curve and should be used if the data system has this capability. Use of $1/X^2$ weighting for linear fits is currently not permitted by method 8000B.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12 hour Calibration

The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The center of each retention time window is updated with each 12 hour calibration.

10.11.2. Calibration Verification

It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12 hour calibration with the exception that retention times are not updated.

10.11.3. Any analyte that is reportable as found must have a % difference of $\leq$ 15% in the preceding verification or 12 calibration, on the column used for quantitation. Refer to section 12.1.2 for which result to report.

10.11.4. Analytes not detected in client samples will ideally also have a %D of $\leq$ 15%. However, the analysis is acceptable if the average of the %D for all the analytes is $\leq$ 15%. This average is calculated by summing all the %D results in the calibration and dividing by the number of analytes.

10.11.5. Reportable as found is defined as any analyte that would be reported as anything other than a non-detect.

10.11.6. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.11.7. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. Bracketing is not necessary for internal standard methods.

10.11.8. % Difference calculation

% Difference for internal and external methods is calculated as follows

Internal Standard:

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard:

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response and calibration factors from the initial calibration

10.11.9. % Drift calculation

% Drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for % drift are the same as for % difference

$$\%\ Drift = \frac{Calculated\ Conc.\ -\ Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.11.10.Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 15% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than ± 15%, a new calibration curve must be prepared.

10.11.11.Corrective Action for Samples

For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be reinjected.

For external standard methods, any samples injected after the last good continuing calibration standard must be reinjected.

11. PROCEDURE

11.1. Extraction

Extraction procedures are referenced in the appendices.

11.2. Cleanup

Cleanup procedures are referenced in the appendices.

11.3. Gas Chromatography

Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

In general, volatiles analytes are introduced using purge and trap as described in Appendix A. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

11.5.1. The daily calibration includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.2. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multiresponse analytes (e.g., Aroclors) use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid point of the initial calibration and each 12 hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than +/- 0.05 minutes, use +/- 0.05 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by the 12 hour calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

The center of the retention time windows determined in section 11.6 are adjusted to the retention time of each analyte as determined in the 12 hour calibration standards. (See the method 8081A and 8082 appendices for exceptions for multi-response components.) The retention time windows

must be updated at the beginning of each analytical sequence and with each 12 hour calibration, but not for any other calibration verification standards.

11.8. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP CORP-OP-0001 for determination of percent moisture.

11.9. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and / or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flag confirmation required).

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst;
A) The primary column approach
Or
B) The better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach:

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary, and the primary column calibration fails, then the sample must be evaluated for reanalysis.)

12.1.2.2. Better result approach

The lower of the two results is normally reported. The lower result is considered better because the higher result is generally higher because of chromatographic interference. The higher result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the lower result
- The continuing or bracketing calibration on the column with the lower result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for reanalysis.)

12.1.3. If the Relative percent difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{R_1 - R_2}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Continuing Calibration

If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.6. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.1. External Standard Calculations

12.6.1.1. Aqueous samples

$$Concentration\ (mg\ /\ L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng, Section 10.1

12.6.1.2. Non-aqueous Samples

$$Concentration\ (mg\ /\ kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\ Moisture}{100}$$ (D = 1 if wet weight is required)

12.6.2. Internal Standard Calculations

12.6.2.1. Aqueous Samples

$$Concentration\ (mg\ /\ L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte

12.6.2.2. Non-aqueous Samples

$$Concentration\ (mg\ /\ kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.3. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated

failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the waste streams detailed in the facility hazardous waste management procedures, Attachment B, Chemical Hygiene Plan, Section WS002, Table 1, current edition.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Section 8000B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Modifications from Previous Revision

This SOP has been substantially revised to reflect changes to Method 8000 contained in Update III to SW-846.

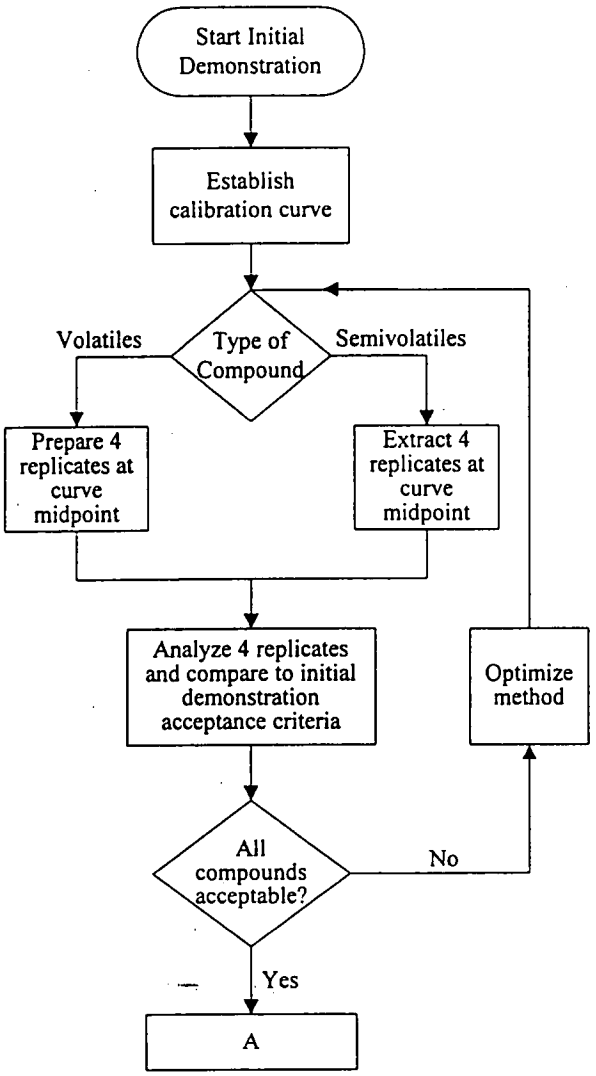
17.3. Facility Specific SOPs

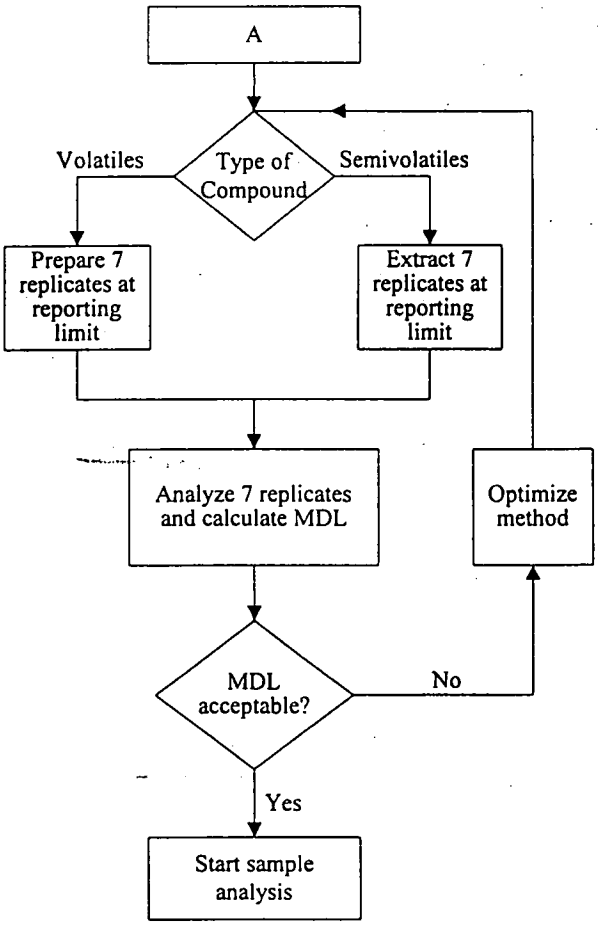
Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Flow Diagrams

17.4.1. Initial demonstration and MDL[1]

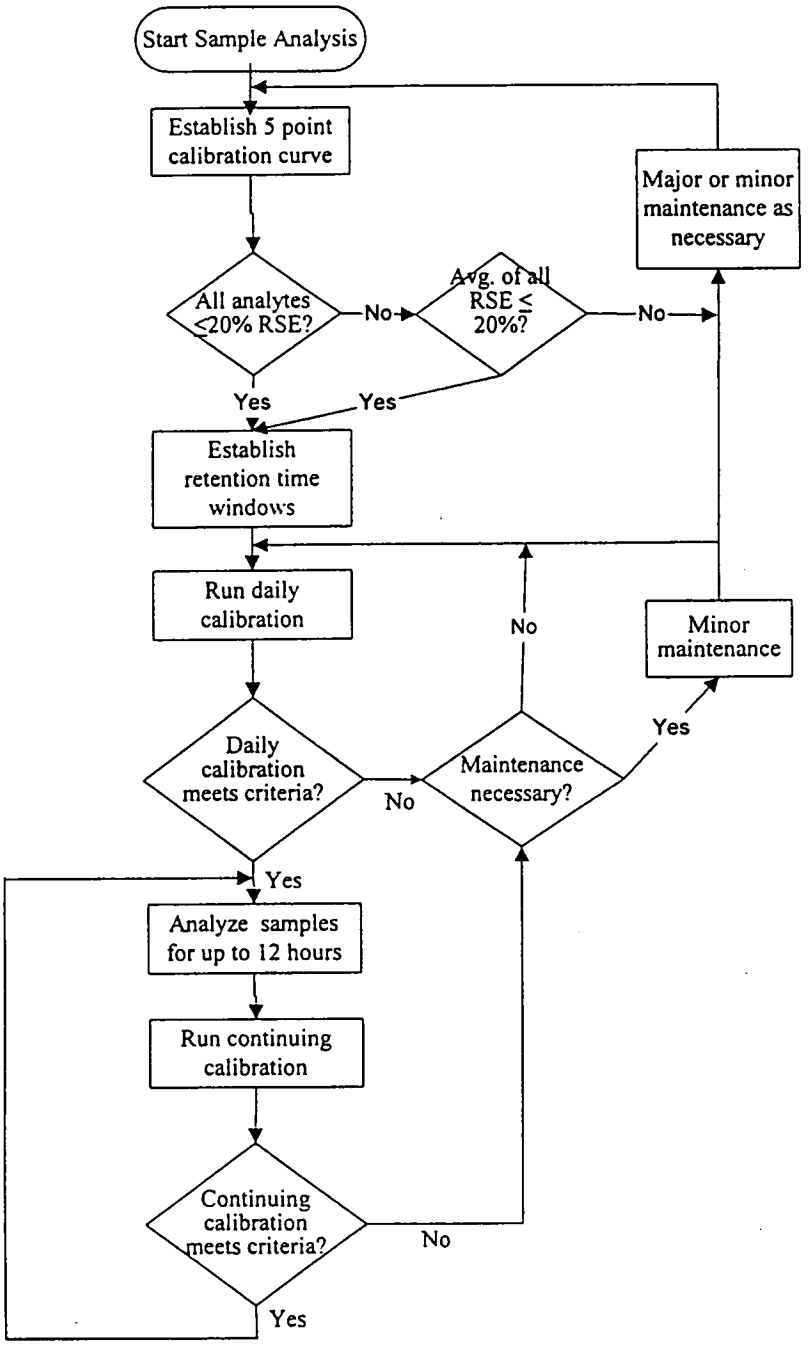
[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.





17.4.2. 17.4.2. Sample Analysis[1]

[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.


Start Sample Analysis
Establish 5 point calibration curve
All analytes ≤20% RSE?
No
Avg. of all RSE ≤ 20%?
No
Major or minor maintenance as necessary
Yes
Yes
Establish retention time windows
Run daily calibration
No
Minor maintenance
Yes
Daily calibration meets criteria?
No
Maintenance necessary?
Yes
Analyze samples for up to 12 hours
Run continuing calibration
Continuing calibration meets criteria?
No
Yes

1. SCOPE AND APPLICATION

1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure, following method 8021B. However, where required by a client QAPP this section may also be used to analyze aromatic volatiles by discontinued methods 8020A and 8010B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are soluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photoionization detector, an electrolytic conductivity detector or a combination of both.

2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method. If very low detection limits are needed for soil samples then direct purge using sodium bisulfate preservation may be necessary.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this SOP.

4. INTERFERENCES

4.1. Refer to section 4 of the method 8000B part of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. When utilizing an autosampler system which has multiple ports for sample analysis, it is likely that only a single stage or port may be contaminated by a highly concentrated sample. If a port is suspect, a water blank should be analyzed to verify lack of contamination. If the water blank and subsequent blanks on that port show contamination consistent with the concentrated sample, further maintenance is required. This may include replacing or cleaning the multi-port valve, transfer lines, etc.

4.6. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 14 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 14 day period prior to the analysis of the contaminated holding blank is required.

4.7. Acidification of samples may result in hydrolysis of 2-chloroethyl vinyl ether.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B section of this SOP for general safety requirements.

5.2. Often, purge vessels on purge-and-trap instrumentation are pressurized by the time analysis is completed. Therefore, vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.3. The toxicity or carcinogenicity of each chemical used in this procedure has not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Methanol -- Flammable and toxic. Methanol readily absorbs via the skin.

Sodium bisulfate--Irritant.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes -- 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.

6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.

6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.7. Columns - Refer to Table A-2 for details of columns.

6.8. Volumetric flasks, Class A: 5 mL to 250 mL

6.9. pH paper

6.10. Balance capable of weighing to 0.01g for samples.

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. Suggested methods for generating organic free water include:

- Filtration through a carbon bed.
- Continuously sparging water with helium or nitrogen.
- Use of commercial water purification systems.

Other methods may be used, so long as the requirement that the water not show any interferences is met. The procedure used should be documented in a lab specific attachment.

7.3. Sodium Bisulfate

7.4. Methanol -- Purge and Trap Grade

7.5. Standards

Refer to tables A-5 and A-6 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.5.1. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take a EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg

for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 12.5 µL of 2000 µg/mL solution for a nominal 25 g sample, 5 µL of a 20 µg/mL solution for a nominal 5 g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 12.5 µL of a 1000 µg/mL solution for a nominal 25 g sample, 5 µL of a 10 µg/mL solution for a nominal 5 g sample.).

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol (Add 12.5 µL of a 1000 µg/mL solution for a nominal 25 g sample, 5 µL of a 10 µg/mL solution for a nominal 5 g sample.).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4±2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a VOA vial by adding 5 mL purge and trap grade methanol. (If a 25 g sample is to be used, add 25 mL methanol to the VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 12.5 µL of 2000 µg/mL solution for a nominal 25 g sample, 5 µL of a 20 µg/mL solution for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 12.5 µL of a 1000 µg/mL solution for a nominal 25 g sample, 5 µL of a 10 µg/mL solution for a nominal 5 g sample.).

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (Add 12.5 µL of a 1000 µg/mL solution for a nominal 25 g sample; 5 µL of a 10 µg/mL solution for a nominal 5 g sample.).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4±2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (methanol preserved) procedure, in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal the vial and attach a label. The label must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

8.7.8. **Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at ≥ -10°C until analysis. Analysis may still proceed utilizing the sodium bisulfate instrument calibration, however, this anomaly must be noted in a nonconformance.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure.

8.8. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

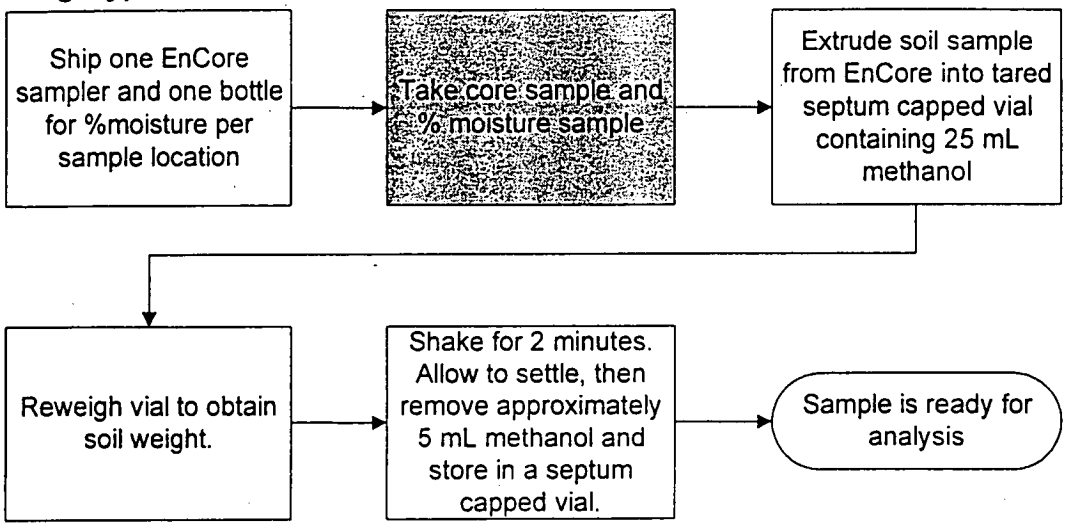
8.9. Medium level solid extracts are aliquoted into 2 - 10 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum

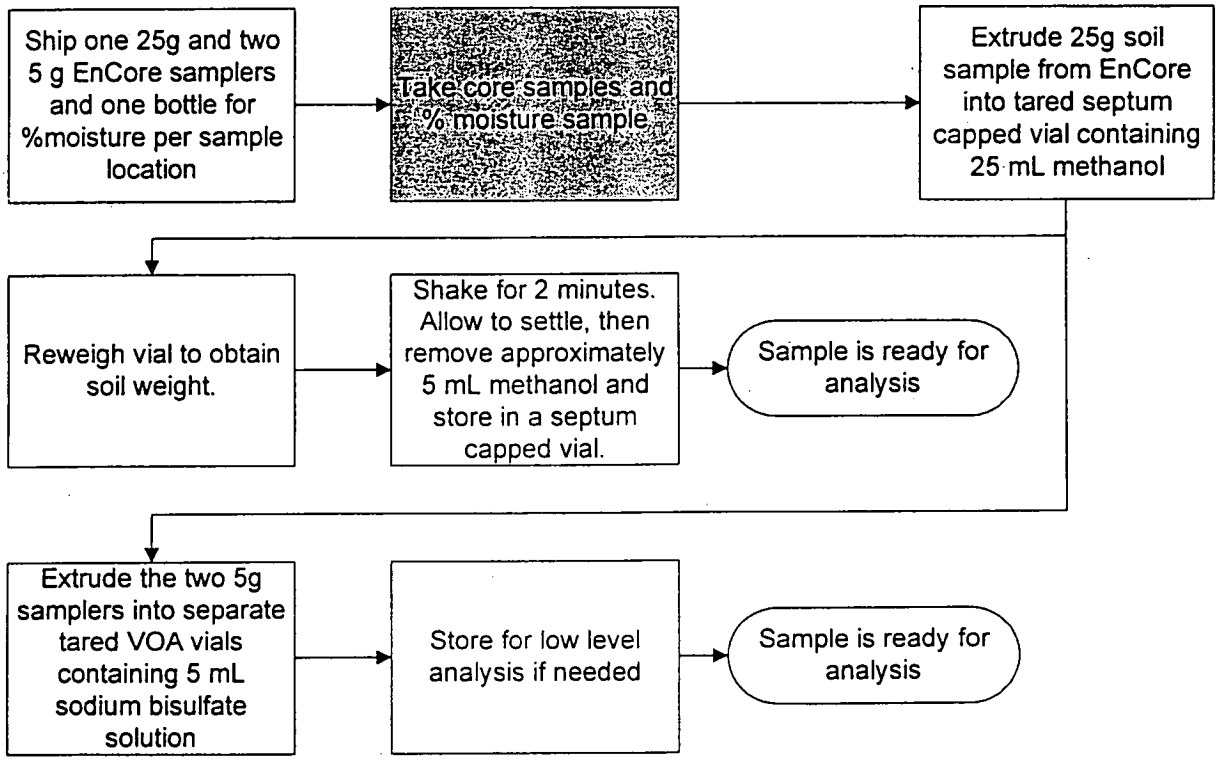
holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.11. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

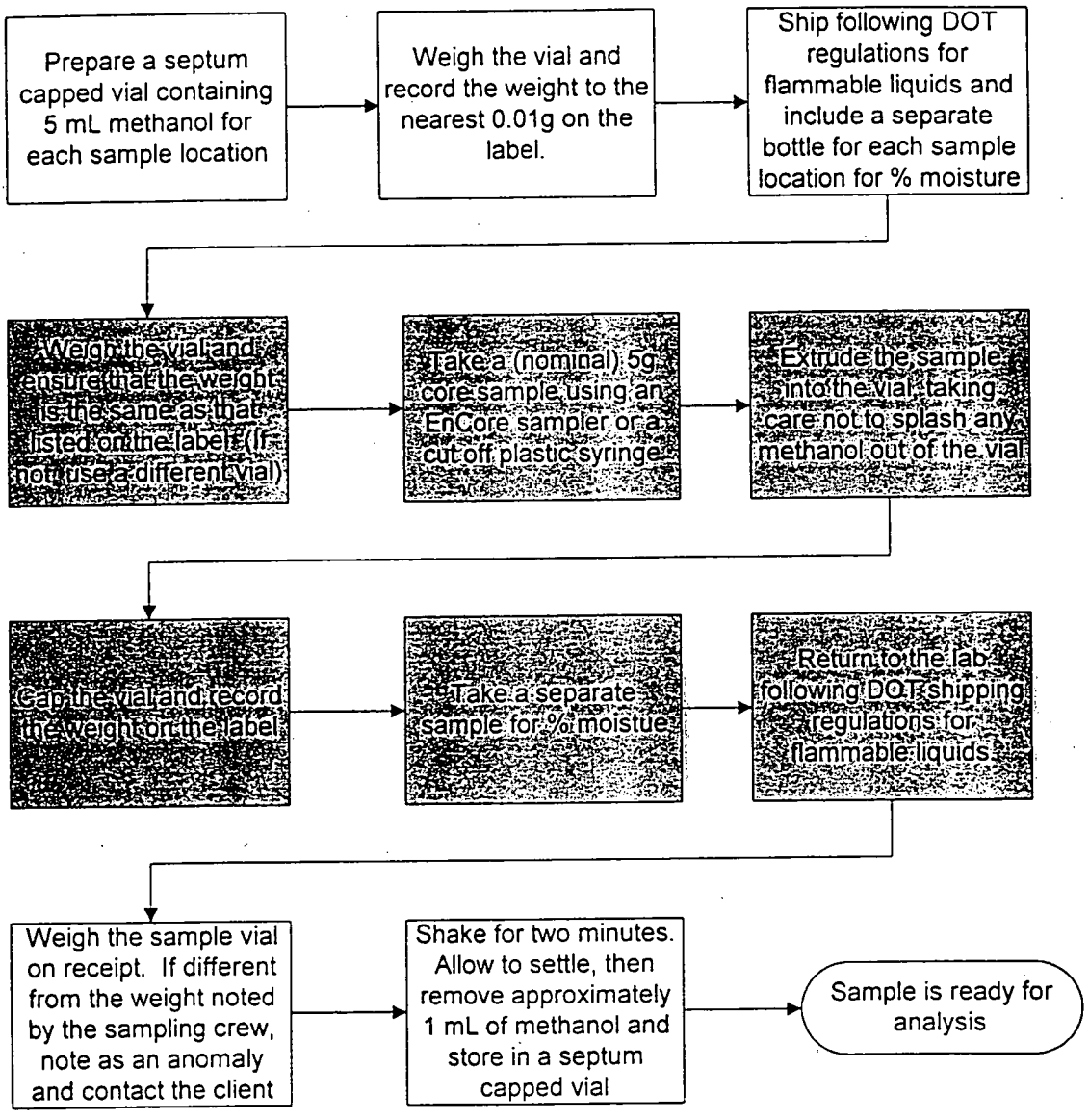
EnCore procedure when low level is not required (field steps in gray)



EnCore procedure when low level is required

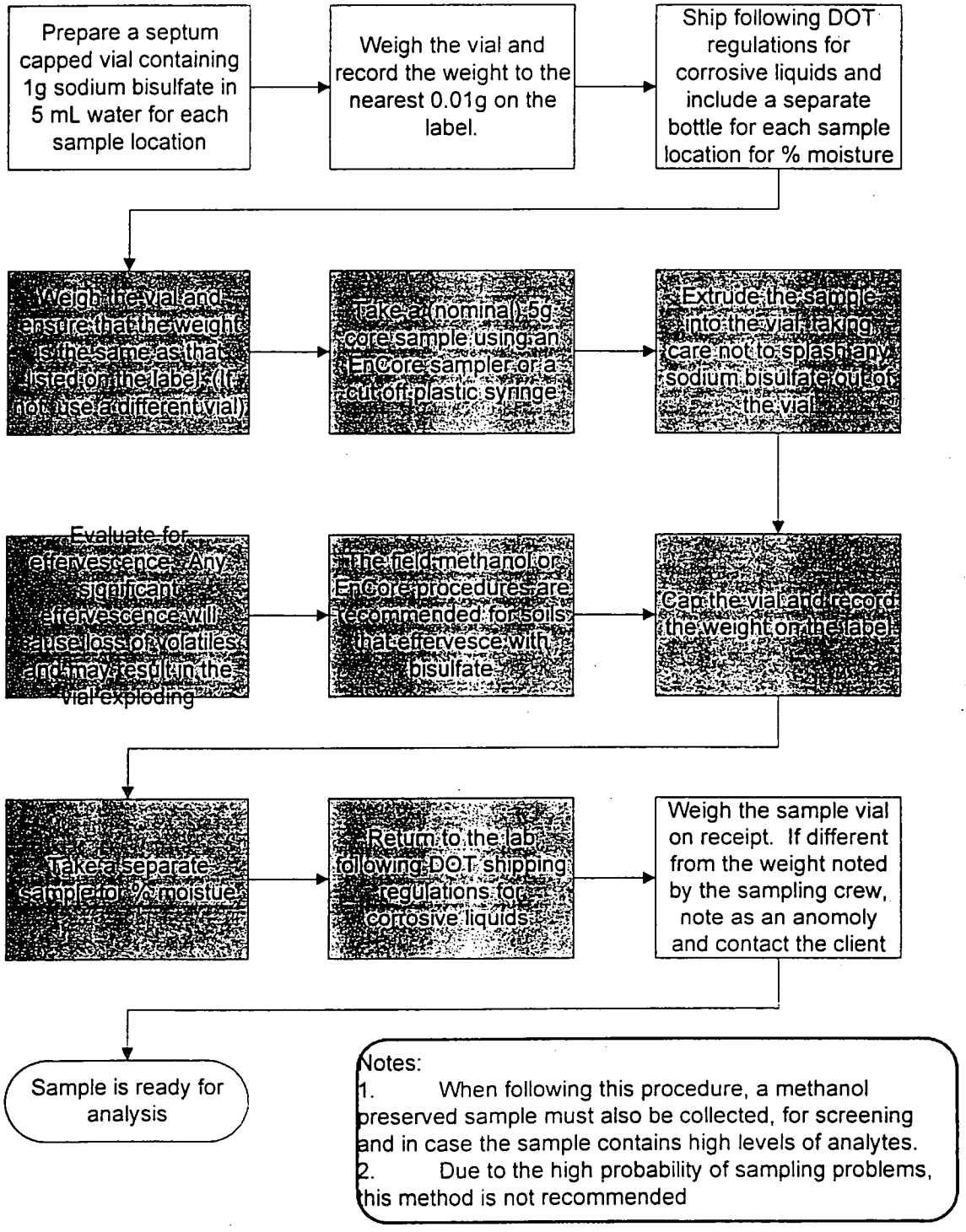


Field methanol extraction procedure (field steps in gray)



9.

Field bisulfate preservation procedure (field steps in gray)



Notes:
1. When following this procedure, a methanol preserved sample must also be collected, for screening and in case the sample contains high levels of analytes.
2. Due to the high probability of sampling problems, this method is not recommended

9. QUALITY CONTROL

9.1. Refer to the method 8000B section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the method 8000B section of this SOP, section 10, for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the 8000B section of this SOP for details of initial calibration criteria.

10.3.2. Low level soil samples must be purged at 40°C, therefore the calibration curve must also be purged at 40°C. In addition, the low level soil calibration solutions should contain approximately the same amount of sodium bisulfate as the samples.

10.3.3. The low level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Calibration Verification

10.4.1. A mid level calibration standard is used for the calibration verification. The gases have a 20%D criteria rather than the 15% used for other analytes.

10.4.2. A calibration verification run is performed after every 10 samples for this method.

10.4.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a 12 hour calibration that meets % difference criteria from an existing initial calibration.

11.3. Confirmation

The PID and ELCD detectors are sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation should be decided in

consultation with the client. If the PID and ELCD are used in series confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

11.4. Aqueous Sample Analysis (Purge and Trap units using sparge vessels)

11.4.1. Depending on the sensitivity of the instrument and capabilities of the purge and trap device, 5, 10, 20, or 25 mL sample volumes may be analyzed. A 5 mL sample volume is recommended.

11.4.2. Rinse a 5 mL (or 25 mL for larger sample volumes) gas-tight syringe with organic free water. Fill the syringe with the sample to be analyzed, and compress to volume.

11.4.3. Check and document the pH of the sample remaining in the VOA vial after loading the syringe.

11.4.4. This procedure invalidates the contents of the VOA vial for further analysis, unless an aliquot is transferred to a smaller VOA vial with no headspace (e.g., 20 mL) at the same time the analysis aliquot is removed.

11.4.5. Spike with the appropriate volume of surrogate/internal standard solution and spike solution (if required) through the barrel of the syringe. The method blank is spiked with surrogates only, the LCS and matrix spikes with the surrogate and matrix spiking solutions. Refer to Tables A-5 and A-6 for volumes and concentrations of spiking solutions.

11.4.6. Load onto the purge and trap device and start the run.

11.4.7. If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample has a high response for a compound, analysis should be followed by an organic free water blank. It is recognized that during automated unattended analysis, this may not occur. If any potential carryover hits are present in samples following highly contaminated samples, the sample must be reanalyzed to determine if any of these hits are a result of carryover or are actually present in the sample.

11.4.8. Dilutions may be made in gas tight syringes unless the volume of sample used is less than 5 µL, in which case dilution in volumetric flasks will be necessary.

11.4.8.1. Spike with the same volume of surrogate/internal standard solution as used for undiluted samples prior to loading onto the purge and trap device.

11.4.8.2. For Matrix spike / matrix spike duplicates where the sample requires dilution, the sample is spiked <u>after</u> the dilution is performed.

11.5. Aqueous and Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.5.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.5.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.5.3. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5.4. Check the pH of the sample remaining in the VOA vial after analysis is completed.

11.6. 11.6. Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.6.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.6.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

11.6.3. Connect the purge vessel to the purge and trap device.

11.6.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.

11.6.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.6.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.6.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect. If external standard calibration is used, samples with surrogate recovery below the control limit should be reanalyzed once to confirm matrix effect.

11.7. Methanol Extract Soils

11.7.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

Refer to section 12 of the 8000B section of this SOP.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000B section of this SOP are 70-130%, with %RSD $\leq$ 25% The spiking level should be 20 µg/L. These limits are for guidance, and certain "difficult" analytes may fall outside this range. Any outliers must be discussed with Quanterra's Director of Quality Assurance and/or Director of Technology before proceeding with sample analysis.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed of according to the waste streams detailed in the facility hazardous waste management procedures, Attachment B, Chemical Hygiene Plan, Section WS002, Table 1, current edition.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.2. Modifications from previous revision

17.2.1. Substantial changes to this method were made to reflect changes in Update III of SW-846.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Tables

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
Halogenated volatiles by 8021B	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chloroethane	70-00-3	1.0	1.0	50
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	Dibromochloromethane	124-48-1	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-45-4	1.0	1.0	50
	cis-1,2 Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	Dichloromethane(DCM)	75-09-2	5.0	5.0	250
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
Additional halogenated volatiles					
	2-Chloroethyl vinyl ether	110-75-8	5.0	5.0	250
	1,2-Dibromoethane	106-93-4	5.0	5.0	250
BTEX by 8021B	Benzene	71-43-2	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	m,p-Xylene		2.0	2.0	100

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	o-Xylene	95-47-6	1.0	1.0	50
Aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	1,2-Dichlorobenzene	75-34-3	1.0	1.0	50
	1,3-Dichlorobenzene	107-06-2	1.0	1.0	50
	1,4-Dichlorobenzene	75-45-4	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	m- & p- Xylene		2.0	2.0	100
	o-Xylene	1330-20-7 95-47-6	1.0	1.0	50
Additional aromatic and unsaturated volatiles					
	Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	1.0	50
Combined halogenated and aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chloroethane	75-00-3	1.0	1.0	50
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	1,2-Dibromoethane(EDB) (Additional Compound)	106-93-4	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50

Table A-1
Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-35-4	1.0	1.0	50
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	Ethylbenzene	100-41-4	1.0	1.0	50
	Methylene Chloride	75-09-2	5.0	5.0	250
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
	m- & p- Xylene		2.0	2.0	100
	o-Xylene	95-47-6	1.0	1.0	50

Table A-2
Recommended Conditions for Aromatic Volatiles

Parameter	Recommended Conditions
Temperature program	40°C, 10min, 8°C/min to 200°C,3min
Column 1	Rtx-624 or DB-624 60m x 0.53mm 3.0um
Column 2	Rtx-1 or DB-1 60m x 0.53mm 3.0 um

Carrier gas	Helium or hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (245°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (260°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3
Recommended Conditions for Method Halogenated Volatiles

Parameter	Recommended Conditions
Temperature program	35°C, 16 min, then 5°C/min to 210°C, hold for 2 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 10 minutes
Desorb Temp / time	180°C, 2 minutes (240°C for Vocarb 3000)
Bake Time / temp	200°C, 15 minutes (270°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-4
Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles

Parameter	Recommended Conditions
Temperature program	35°C, 16 min, then 5°C/min to 210°C, hold for 2 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 10 minutes
Desorb Temp / time	180°C, 2 minutes (240°C for Vocarb 3000)
Bake Time / temp	200°C, 15 minutes (270°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-5
Surrogate and Internal Standard Concentrations

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic volatiles IS/SS			5	
		20		20
	1-Chloro-4-fluorobenzene (IS)	20		20
	a,a,a-Trifluorotoluene (SS)			
	Bromochloromethane (SS)	20	5	20
	1-Chloro-3-fluorobenzene (IS)	20		20
Combined Aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	20	5	20
	Bromochloromethand (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20		20
	1-Chloro-3-fluorobenzene (IS)	20	5	20

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometimes be requested as a target analyte. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane: Elutes very close to chloroform and cis-1,2-dichloroethene on the 502.2 column. May be a target analyte.
1,2-Bromochloroethane:
1-Chloro-2-fluorobenzene: Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2
a,a,a-Trifluorotoluene: Good for aromatic volatiles, coelutes or very close to trichloroethene
Bromofluorobenzene: Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.
2-Bromo-1-chloropropane: May coelute with 1,1,2-trichloroethane

Table A-6				
Concentrations for LCS and MS/MSD compounds				
Standard	Components	Working Solution µg/mL	Spike amount µL (5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic	Benzene	10	5	10
	Toluene	10		10
	Chlorobenzene	10		10
Halogenated	Chlorobenzene	10	5	10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10
Combination aromatic / halogenated	Benzene	10	5	10
	Toluene	10		10
	Chlorobenzene	10		10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix may also to be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate Quanterra sample extraction SOPs. (CORP-OP-0001)

Table B-1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

At client request, this method may also be used for the analysis of PCBs (Arochlors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. In any event, if samples for PCB analysis do not need the acid clean up procedure, then the same injection may be used for method 8081B and 8082, assuming all calibration and QC requirements for both methods are met. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation may be by internal or external standard methods.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001. Use or hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

Any contact must be reported to the Area Leader and to Environmental Health and Safety for assessment.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to tables B-5 and B-6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Refer to Table B-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns. Closely eluting pairs are DDE and dieldrin on the RTX-5 or DB-5 column, and Endosulfan II and DDD on the DB-1701 column.

10.3. Column Degradation Evaluation

Before any calibration runs, either initial or 12 hour, the column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of a capillary column.
- Replacing the GC column.

10.4. Initial Calibration

Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

10.4.1. Refer to Table B-8 for the initial calibration analytical sequence.

10.4.2. The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

10.4.3. The surrogate calibration curve is calculated from the Individual AB mix. Surrogates in the other calibration standards are used only as retention time markers. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.4.4. For multi-component pesticides:

Single point calibration is used for multicomponent pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 12.3 for guidance on which option to use.

10.4.5. For multicomponent analytes, the mid level standard must be analyzed as part of the initial calibration. This single point calibration is used to quantitate multicomponent analytes.

10.4.6. The analyst may include a full 5 point calibration for any of the multicomponent analytes with the initial calibration.

10.5. 12 hour Calibration Verification

The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

10.5.1. At a minimum, the 12 hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multicomponent analytes.

10.5.2. The retention time windows for any analytes included in the 12 hour calibration are updated.

10.6. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multicomponent analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

10.6.1. A mid level calibration standard is used for the continuing calibration.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

11.4. Suggested gas chromatographic conditions are given in Table B-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table B-8.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multicomponent Analytes

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multicomponent Analytes

Use 3-10 major peaks or total area for quantitation as described in section 10.4.4, initial calibration of multicomponent analytes.

12.3.1. If there are no interfering peaks within the envelope of the multicomponent analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.1.1. Multiple peak option

This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

Chlordane may be quantitated either using the multiple peak option (12.3.1.1) total area option (12.3.1.2.) or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.3.1.2. Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

12.4. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

$$DDT\,\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

where:
A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

$$Endrin\,\%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

where:
A_{EK}, A_{EA}, and A_{E} = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP are presented in Table B-7. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed of according to the waste streams detailed in the facility hazardous waste management procedures, Attachment B, Chemical Hygiene Plan, Section WS002, Table 1, current edition.

16. REFERENCES

SW846, Update III, December 1996, Method 8081A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

None

17.2. Modifications from Previous Revisions

17.3. Substantial revision were made to reflect the changes to method 8081 in Update III to SW-846.

17.4. Tables

Table B-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.5	17	500
Toxaphene	2.0	67	2000
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzillate	0.1	3.3	100
Additional ADD-ONs			
2,4'-DDE	0.10	3.3	100
2,4'-DDD	0.10	3.3	100
2,4'-DDT	0.10	3.3	100
Chlordane (technical)	0.5	17	500

Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects. Reporting Limits may be lowered in the future for some compounds as an appropriate custom mix becomes available.

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table B-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	150°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	DB-608 30m X 0.53 mm, 0.83 µm
Column 2	DB-1701 30m X 0.53mm 1.0 µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	2.5	5	10	10	50	100
g-BHC (Lindane)	2.5	5	10	10	50	100
Heptachlor	2.5	5	10	10	50	100
Methoxychlor	25	50	100	200	500	1000
Dieldrin	5	10	25	50	100	200
Endosulfan I	2.5	5	10	20	50	100
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	2.5	5	10	20	50	100
δ-BHC	2.5	5	10	20	50	100
α-BHC	2.5	5	10	20	50	100
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane[3]	2.5	5	10	20	50	100
γ-Chlordane[3]	2.5	5	10	20	50	100
Multicomponent Standards						
Chlordane (Technical)			250[4]			
Toxaphene			1000[5]			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	2.5	5	10	20	50	100
Decachlorobiphenyl	5	10	20	40	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.
[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.
[4] This standard may be used for quantitation of technical chlordane between 50 and 1000 ng/mL. If the chlordane is more concentrated, the extract must be diluted and reanalyzed.
[5] This standard may be used for quantitation of toxaphene between 200 and 4000 ng/mL. If the toxaphene is more concentrated, the extract must be diluted and reanalyzed.

Table B-4	
Column Degradation Evaluation Mix ng/mL	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	16.67	200
Aldrin	0.20	16.67	200
Heptachlor	0.20	16.67	200
Dieldrin	0.50	33.3	500
Endrin	0.50	33.3	500
4,4'DDT	0.50	33.3	500
Tetrachloro-m-xylene (Surrogate)	0.20	13.3	200
Decachlorobiphenyl (Surrogate)	0.20	13.3	200

Table B-6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP µg/L or µg/kg		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-7
Suggested Analytical Sequence

Initial Calibration

Solvent blank (optional)	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
Solvent blank	
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	
Any multicomponent analytes	

[1] A five point curve for any of the multicomponent analytes may be included
If Aroclors are included, a 5 point calibration for Aroclor 1016/1260 should be included with the initial calibration and a single point for the other Aroclors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

Table B-8		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
Aldrin	46-112	21
alpha-BHC	51-122	24
beta-BHC	61-120	32
delta-BHC	49.5-118.5	36
gamma-BHC	57-116	23
Chlordane	44.8-108.6	20
4,4'-DDD	52-126	28
4,4'-DDE	46-120	27.5
4,4'-DDT	54-137	36
Dieldrin	42.5-124.5	38
Endosulfan I	43-141	24.5
Endosulfan II	78-171	61
Endosulfan Sulfate	62-132	27
Endrin	49-126	37
Heptachlor	57-100	20
Heptachlor Epoxide	43.5-131.5	25.4
Toxaphene	44.4-111.2	20

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate Quanterra sample extraction SOPs. (CORP-OP-0001). The PCBs are determined and quantitated as Arochlor mixes.

Table C-1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly. Any contact must be reported to the Area Leader and Environmental Health and Safety for assessment.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. All standards for this method must be replaced

7.2. Refer to Table C-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Other surrogates may be used at client request. Refer to Table C-4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Initial Calibration

10.2.1. Refer to Table C-5 for the initial calibration analytical sequence.

10.2.2. The response for each Arochlor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

10.2.3. A five point calibration of the Aroclor 1016/1260 mix is generated with at least mid level single points for the other Aroclor mixes. The average response factor is used to quantitate Aroclors 1260 and 1016, other Arochlors are quantitated from the mid level single point of the individual Aroclor.

10.2.4. The analyst may include a full 5 point calibration for any of the Aroclors with the initial calibration.

10.2.5. The high and low standards for the initial 5 point calibration of 1016 / 1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration the extract must be diluted and reanalyzed.

10.2.6. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the five point calibration rather than the 1016 / 1260 mix.

10.2.7. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.8. Two options are possible for quantitation of Aroclors. The same quantitation option must be used for standards and samples.

10.2.8.1. Multiple peak option

Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per Aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

10.2.8.2. Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

10.3. 12 hour Calibration

The 12 hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.

10.3.1. At a minimum, the 12 hour calibration includes analysis of the Aroclor 1016/1260 mix.

10.3.2. It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific Aroclor is expected, it should be included in the daily calibration check.

10.3.3. The retention time windows for any analytes included in the daily calibration are updated.

10.3.4. For this method samples must be bracketed with successful calibration verification runs.

10.4. Calibration verification

The Aroclor 1016/1260 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

10.4.1. A mid level standard is used for the calibration verification.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

11.4. Suggested gas chromatographic conditions are given in Table C-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C-5.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

A clearly identifiable Aroclor pattern serves as confirmation of single column GC analysis. However, if the pattern is not clear, or if no historical data for the site is available, then second column confirmation must be performed.

12.2. Quantitation of Aroclors

Use 3-10 major peaks or total area for quantitation

If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant Aroclor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well separated Aroclor patterns are present, then both Aroclors are quantitated and reported.

12.3. If there are no interfering peaks within the envelope of the Aroclor, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.4. Second column confirmation of Aroclors will only be performed when requested by the client, or if the pattern is not clear or there is no historical data leading to a suspicion that Aroclors may be present. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP are recovery of 70-130%. The spiking level should be equivalent to a mid level calibration.

13.2. Method detection limits (MDL) are determined for Aroclor 1016 and 1260. Aroclor 1016 represents an Aroclor consisting primarily of low chlorinated congeners while Aroclor 1260 represents an Aroclor consisting primarily of high chlorinated congeners. The same reporting limit is applied to all Aroclors, and must be supported by both MDLs.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed o faccording to the waste streams detailed in the facility hazardous waste management procedures, Attachment B, Chemical Hygiene Plan, Section WS002, Table 1, current edition.

16. REFERENCES

SW846, Update III, December 1996, Method 8082

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. Modifications from Previous Revisions

The 8082 procedure was added to this revision.

17.3. Tables

Table C-1 Standard Analyte list and Reporting Limits			
	Reporting Limit, µg/L or µg/kg		
Compound	water	soil	waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table C-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[1]
Aroclor 1016/1260	50	100	200	300	500	1000
Aroclor 1242[2]			200			
Aroclor 1221 +1254[2]			200			
Aroclor 1232[2]			200			
Aroclor 1248[2]			200			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	30	50	100	200
Decachlorobiphenyl	5	10	30	50	100	200

[1] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[2] Aroclors may be quantitated within the range 50 to 500 ng/mL (1000ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table C-4			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup µg/L or µg/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	2	167	2000
Tetrachloro-m-xylene (Surrogate)	0.20	13.3	200
Decachlorobiphenyl (Surrogate)	0.20	13.3	200

Table C-5
Suggested Analytical Sequence

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	Level 3
8	Aroclor 1242	Level 3
9	Aroclor 1248	Level 3
10	Aroclor 1221/1254	Level 3
11	Solvent blank	
12-31	Sample 1-20 (or as many samples as can be analyzed in 12 hours	
	Solvent blank (optional)	
32	Aroclor 1016/1260	Level 3

etc

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1016/1260 mix. Mid level standards of any other Aroclors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP CORP-OP-0001. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in section 9 and the initial demonstration of method performance in section 13 are met.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Sulfur may interfere and may be removed by the procedure described in SOP#CORP-OP-0001.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table D-3 and D-4 for details of calibration and other standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D-5 for minimum performance criteria for the initial demonstration of capability.

9.3. Refer to Table D-4 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method.

10.3. A mid level standard is used for the continuing calibration.

10.4. Refer to Table D-2, for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001.

11.3. Cleanup

The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration must be analyzed at least once every 12 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

Chromatographic conditions are listed in Table D-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L

calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

13. METHOD PERFORMANCE

13.1. Multiple laboratory performance data has not been published by the EPA for this method. Table D-5 lists minimum performance standards required by Quanterra for the four replicate initial demonstration or capability (required by Section 13.2 of the 8000B part of this SOP) for this method. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed of according to the waste streams detailed in the facility hazardous waste management procedures, Attachment B, Chemical Hygiene Plan, Section WS002, Table 1, current edition.

16. REFERENCES

Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

Refer to the method 8000B section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

Modifications to the calibration directions were made in order to make the method consistent with method 8000B.

17.3. Tables

Table D-1				
Standard Analyte list				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL	10 mL	20
Low-level Soil without GPC	50 g	10 mL	20
High-level soil / waste	1 g	10 mL	20

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D-1.

Table D-2	
Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5um
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table D-1.

Table D-4			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg[1]			
	Aqueous	Soil	Waste
2,4-D	16	800	16000
Silvex	4	200	4000
2,4,5-T	4	200	4000
2,4-DB	16	800	16000
Dalapon	8	400	8000
DCAA (surrogate)	16	800	16000

[1] LCS, MS and SS spikes are as the free acid.

Table D-5		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
2,4-D	50-150	25
2,4-DB	50-150	25
2,4,5-TP (Silvex)	50-150	25
2,4,5-T	50-150	25
Dalapon	50-150	25
Dicamba	50-150	25
Dichloroprop	50-150	25
Dinoseb	25-120	40
MCPA	50-150	25
MCPP	50-150	25

The reporting limits listed in Table D-1 will be achieved with these calibration levels and a 20 fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D-3						
Calibration Standards, free acid equivalent (methyl ester)[1] ng/mL						
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
2,4-D	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)	640 (681)
2,4-DB	20 (21.1)	40 (42.2)	80 (84.5)	160 (169)	320 (338)	640 (676)
2,4,5-TP [Silvex]	5 (5.25)	10 (10.5)	20 (21.1)	40 (42.1)	80 (84)	160 (168)
2,4,5-T	5 (5.27)	10 (10.5)	20 (21.1)	40 (42.2)	80 (84.4)	160 (169)
Dalapon	10 (11.0)	20 (22.0)	40 (43.9)	80 (87.8)	160 (176)	320 (351)
Dicamba	10 (10.6)	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)
Dichloroprop	20 (21.2)	40 (42.4)	80 (84.8)	160 (170)	320 (339)	640 (678)
Dinoseb	3 (3.17)	6 (6.35)	12 (12.7)	24 (25.4)	48 (50.8)	96 (102)
MCPA	2,000 (2,140)	4,000 (4,280)	8,000 (8,560)	16,000 (17,100)	32,000 (34000)	64,000 (68,000)
MCPP	2,000 (2,120)	4,000 (4,260)	8,000 (8,520)	16,000 (17,000)	32,000 (34,100)	64,000 (68,200)
2,4-DCAA [Surrogate]	20	40	80	160	320	640

[1] The calibration standard solutions contain the herbicides as their methyl esters at the levels in parentheses above. These levels are equivalent to the free acid levels also listed above. The herbicides are reported as the free acids.

[2] Level 6 is optional

ATTACHMENT C

QUANTERRA QUALITY CONTROL PROGRAM

APPROVAL:

Director, Quality Assurance Services

Vice President, Operations

Quanterra Incorporated®

COMPANY-WIDE

Rev. 2, 07/13/98
Supersedes: Revision 1

POLICY NAME

Quanterra® Quality Control Program

Policy #: QA-003

OBJECTIVE:

This policy describes the Quanterra® program of routine analytical quality control (QC) activities. The objective is to generate QC data that demonstrate that the analytical process is in control and that the data meet client and method requirements.

SCOPE:

This policy is to be enforced and followed throughout the company.

POLICY:

1.0 Assessments of QC data relative to control limits determine the acceptability of sample test results. Whenever control criteria are not met, the data must be evaluated to determine appropriate corrective action. The initial evaluation is made by the analyst, frequently in

conjunction with data review software and /or senior analysts or supervisors. Further technical evaluation of the data or data review software output is conducted by second-party data reviewers. Corrective action decisions, particularly whether or not to reanalyze samples, should be done in consultation with the client to the extent possible when operating under project-specific QA plans. Requirements for assessment and corrective action are described in the attachments to this policy. Details concerning technical data review and documentation of the reviews are described in "Technical Data Review Requirements" (Policy QA-012).

2.0 Quanterra's® standard QC program is to be communicated to the client prior to acceptance of work. At the same time, every effort must be made to understand clients' special project requirements. Generally, laboratory project managers serve as a liaison between the clients and the laboratory staff to ensure that requirements are properly communicated in writing to both parties. In the event that alternative QC procedures are not specified by our clients, these standard QC protocols must be followed to ensure the generation of legally and scientifically defensible analytical data.

3.0 Successful implementation of this QC program requires that it is clearly understood by all Quanterra® staff. Training based on this policy will be conducted at each Quanterra® facility and provided to new personnel as appropriate for their functions.

4.0 Quanterra's® QC program applies to the following regulatory programs:

4.1 RCRA and SW-846 Methods - All routine analytical projects performed using SW-846 methods must comply with the requirements described in Quanterra's® Quality Assurance Management Plan (QAMP, Tables 8.4-2 through 8.4-6) and Attachment I to this policy. The Quality Control sections of analytical standard operating procedures (SOPs) referencing SW-846 methods must be consistent with the requirements in Attachment I

4.2 CWA and 40 CFR Part 136 Listed Methods - Any analytical work conducted in support of an NPDES permit or other Clean Water Act compliance activities, must meet the quality control specifications shown in Quanterra's® QAMP tables 8.4-2 through 8.4-6. The quality control requirements for the specific methods listed in the QAMP define the minimum requirements that must be given in laboratory analytical SOPs.

4.3 CERCLA and CLP Methods - Projects performed using USEPA Contract Laboratory Program (CLP) methods must comply with the quality control specifications shown in Quanterra's® Quality Assurance Management Plan (QAMP) table 8.4-7. The quality control sections of the SOPs referencing CLP protocols must be consistent with the QAMP and the relevant CLP Statement of Work.

4.4 Other Programs or Projects with Clearly Defined QC Requirements

4.4.1 The differences between Quanterra's® standard QC program and special project requirements must be specified in project documents. These documents may include Quality Assurance Project Plans (QAPjPs), Quality Assurance Program Plans (QAPPs), Sampling and

Analysis Plans (SAPs), project-specific Quality Assurance Summaries (QASs), SOPs, contracts, or other approved documents.

4.4.2 Documents describing special project requirements must be reviewed and approved by appropriate QA and operations staff.

4.4.3 If the special project requirements appear to result in modifications that contradict federal or state regulatory requirements, the variance must be noted in writing and communicated to the client. A record of this communication must be retained as a permanent part of the project file.

4.4.4 Any special client's project requirements must be communicated to Quanterra's® analysts in advance of releasing samples for analysis, and the work must be clearly differentiated in the analytical documentation, otherwise Attachment I requirements will be followed.

4.5. Projects Without Specific QC Requirements
Any projects for which no specific QC program is specified, regardless of the source of the analytical methods being used, must follow the requirements shown in Attachment I.

4.6 Radiochemistry QC

General QC requirements for radiochemistry analyses are provided in Attachment II.

ATTACHMENT I
QUANTERRA® QUALITY CONTROL PROGRAM

1.0 INTRODUCTION

This quality control (QC) program is based on the requirements in "Test Methods for Evaluating Solid Waste", USEPA SW-846, Third Edition with promulgated updates. It applies whenever SW-846 analytical methods are used. It also applies in whole or in part whenever project requirements fail to specify some aspect of QC practices described here. It does not apply when other well defined QC programs (e.g. CLP, CLP-like, AFCEE, ACOE, or NFESC) are specified. This policy represents Quanterra's® base QC program for environmental analyses.

Details concerning instrument calibrations, tunes, and QC that are required for specific methods (e.g., interference check samples for ICP and isotopic spikes for dioxin procedures) are not given here. Refer to the method standard operating procedures (SOPs) for information about the frequency, assessment and corrective action required for additional QC elements.

2.0 DEFINITIONS

2.1 QC Batch - The QC batch is a set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and that are processed within the same time period using the same reagent and standard lots.

2.2 Surrogates - Surrogates are organic compounds similar in chemical behavior to the targetanalytes, but that are not normally found in environmental samples. Surrogates are added to all samples in a batch to monitor the effects of both the matrix and the analytical process on accuracy.

2.3 Method Blank - The method blank (MB) is a control sample prepared using the same reagents used for the samples. As part of a QC batch, it accompanies the samples through all steps of the analytical procedure. The method blank is used to monitor the level of contamination introduced to a batch of samples as a result of processing in the laboratory.

2.4 Instrument/Calibration Blank - The instrument blank is prepared using the same solvents and reagents (e.g. hexane, methylene chloride, or reagent water) used to dilute the prepared sample extracts or digests. Unlike the method blank, it is analyzed without being subject to the preparation steps of the analytical procedure. It is used to monitor laboratory or reagent contamination introduced at the instrumental analysis phase of work. For procedures without a separate preparation step, an instrument blank is equivalent to the method blank, and serves the same purpose.

2.5 Laboratory Control Sample - A laboratory control sample (LCS) is prepared using a well characterized matrix (e.g. reagent water or Ottawa sand) that is spiked, with known amounts of representative analytes. Alternate matrices (e.g. glass beads) may be used for soil analyses when Ottawa sand is not appropriate. As part of a QC batch, it accompanies the samples through all steps of the analytical process. The LCS is used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix.

2.6 Matrix Spike and Matrix Spike Duplicate

2.6.1 Matrix Spike - A matrix spike (MS) is a replicate portion of one field sample in the QC batch that is spiked with known amounts of target analytes. An MS is spiked with the same analytes at the same concentrations that are added to the LCS. As part of the QC batch, it accompanies the field samples through all steps of the analytical process.

2.6.2 Matrix Spike Duplicate - A matrix spike duplicate (MSD) consists of an additional portion of the same sample used to prepare the MS. This portion is spiked and processed exactly as is the MS.

2.6.3 The MS and MSD results are used to determine the effect of the sample matrix on the precision and accuracy of results. Due to the potential variability of the matrix of each sample, the MS and MSD results may not have immediate bearing on any samples except the one spiked.

2.7 Sample Duplicate - A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

2.8 Duplicate Control Sample - Duplicate laboratory control samples (LCS/LCSD, also called a DCS pair) consist of a pair of LCSs analyzed within the same QC batch to

monitor precision and accuracy independent of sample matrix effects. This QC sample is prepared at the request of the client and/or when insufficient sample volume is received to prepare and analyze an MS/MSD pair. DCS is intended to provide information regarding the precision of the measurement process.

3.0 BATCH QC ELEMENTS & BATCH PROCESSING

3.1 A QC batch is designed to determine the quality of the analytical results obtained for a group of up to 20 field samples in terms of accuracy and precision. With some exceptions as described in Sections 3.6 through 3.8 below, the minimum QC elements for each QC batch are

- one method blank (MB),
- one laboratory control sample (LCS),
- one matrix spike (MS), and
- one matrix spike duplicate (MSD).

3.2 The identity of each QC batch must be documented and traceable, i.e., each batch of field samples must be clearly associated with the applicable QC samples.

3.3 To the extent possible, samples that require a preparation step should be analyzed together with their associated QC samples. If the samples in a given QC batch require separate analytical runs, the minimum batch QC in each run is an acceptable MB or instrument/calibration blank. To the extent possible, the QC samples should not be analyzed independent of the field samples on a different instrument.

3.4 For analytical procedures that do not include a separate extraction or digestion (e.g., volatile organic analysis by purge and trap), the QC batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

3.5 Field QC samples (e.g., trip blanks, equipment rinsates, and field duplicates) count as individual samples, therefore, they add to the QC batch count. Samples that require simple reanalysis (e.g., dilutions to adjust a sample extract to the working range of the instrument), as opposed to reextraction or digestion and reanalysis, do not count as additional samples in the QC batch. For procedures without a separate preparation, a reanalysis within the same calibration event (as defined in Section 3.4) does not add to the batch count.

3.6 MS/MSD pairs are not the only acceptable means of demonstrating precision.

3.6.1 As requested by clients or required by some methods, batch precision may also be demonstrated through the analysis of sample duplicates (DUs). However, the client should be advised that a DU is less likely to provide usable precision statistics depending on the likelihood of finding concentrations below reporting limits.

3.6.2 A duplicate LCS (DCS) may be used to demonstrate method batch precision independent of client's matrix when the client has not supplied sufficient sample quantity to prepare an MS, MSD or DU, if requested by the client.

3.6.3 On-going monitoring of LCS results can be used to determine long-term (a.k.a., between batch) precision and accuracy for a method.

3.7 Some methods including isotope-dilution methods, pH and ignitability for example, do not use all of the QC elements listed in Section 3.1. Method exceptions to these requirements are listed in the QC tables of Quanterra's® QAMP and in the laboratory's analytical SOPs.

3.8 Deviations from these QC elements must either be noted in project planning documents (QAPPs, QAPjPs, SAPs, SOWs, QAS, or equivalent) or in a NCM.

4.0 DATA EVALUATION AND CORRECTIVE ACTION

4.1 GENERAL GUIDELINES

4.1.1 Any QC component that is outside of established control limits is considered an out-of-control event. All out-of-control events must be documented and the associated data evaluated. Depending on the specific circumstances, evaluation can lead to a variety of actions. The following sections and the flowcharts describe the appropriate corrective action for the most common QC failures. However, it is not possible to address all possible data evaluation scenarios in this policy. The guiding principle for all evaluations is that the data and corrective action decisions must be defensible using Quanterra® policies, procedures or scientific evidence, and justified in the project records.

4.1.2 If reanalysis for QC failures is conducted and the second analysis confirms a QC problem that is outside of the laboratory's control, further testing is not necessary. The problem must be documented and the data properly qualified in the project report.

4.1.3 QC failures that are not corrected by reanalysis are documented in NCMs or using an electronic anomaly system as described in SOP # CORP-QA-0010. This does not include QC failures due to sample matrix interferences (particularly MS, MSD and surrogate failures). These failures are a function of the method used and are normally outside of the laboratory's control; trending via NCMs is not required. Other forms (for example, data review checklists) may be used to document matrix QC failures.

4.1.4 When ongoing, systematic problems are identified, work must stop until it can be demonstrated that the system is in control again.

4.2 Method Blank (MB) Evaluation (also see Figure 1)

4.2.1 Acceptance Criteria
All analyte concentrations in the MB must be less than or equal to the project reporting limit.

4.2.2 Corrective Action for MB Failure
The general corrective action requirement is first reanalysis of the MB, and if not resolved then repreparation and reanalysis of all samples in the QC batch.

The following special situations, while requiring documentation, may allow qualified data to be reported without reanalysis:

- method blank contaminant analyte concentration found in samples is less than the RL,
- analyte concentration in samples is greater than 20 times MB concentration, or
- the analyte is a common laboratory contaminant (see below) and the MB concentration is less than five times the RL for organic analyses or less than two times the RL for inorganic analyses.

Common Laboratory Contaminants:	Method
Methylene Chloride	Volatile Organics (GC or GC/MS)
Acetone	Volatile Organics (GC or GC/MS)
2-Butanone	Volatile Organics (GC or GC/MS)
Phthalate Esters	Semi-Volatile Organics (GC or GC/MS)
Copper	Metals (ICP or GFAA)
Zinc	Metals (ICP or GFAA)
Iron	Metals (ICP or GFAA)
Lead	Metals (Trace ICP or GFAA)

4.3 Laboratory Control Samples (LCS) Evaluation (also see Figure 2)

4.3.1 Acceptance Criteria

The LCS recovery for the control analytes must be within established control limits. The percent recovery is calculated as follows:

$$\textbf{\textit{LCS Percent Recovery}} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of spike added

4.3.2 Corrective Action for LCS Failure

- check calculations,
- check instrument performance,
- reanalyze the LCS, and if still outside of control limits,
- reprepare and reanalyze all samples in the QC batch.

Notes:

Notes:

1. It is acceptable to report the data, if the LCS recovery is out high and analyte of concern was not detected in any of the samples. Successful MS/MSD and surrogate recoveries may also provide evidence of control of accuracy, but this would be decided on a case-by-case basis and only with client approval.

2. In the case of volatile analyses, if the LCS fails, it may be reprepared and reanalyzed within the same tune period.

3. In the case where all target requested analytes are within control, but some other LCS compounds are out of control, the LCS may still be considered acceptable for reporting.

4.4 DUPLICATE LABORATORY CONTROL SAMPLES (LCS/LCSD OR DCS) EVALUATION (ALSO SEE FIGURE 2)

4.4.1 Acceptance Criteria

The recovery for each spike of the pair must be within established control limits. The formula used to calculate LCSD recoveries is the same as the formula for LCS spike recoveries. If a batch includes samples requiring LCS control and samples requiring DCS control, the LCS used will be the first LCS that passes control criteria.

The relative percent difference (RPD) for the pair is calculated as follows:

$$\text{RPD} = \left[\frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}}\right] \times 100$$

Where: X_1 = first observed concentration

X_2 = second observed concentration

4.4.2 Corrective Action for LCS/LCSD Recovery (Accuracy) Failure

- check calculations,
- check instrument performance,
- reanalyze and/or reprepare and reanalyze all samples in the QC batch.

Note: If either LCS/LCSD spikes fails and the batch cannot be reanalyzed, the failure must be documented and noted in the final report.Also see notes under Section 4.3.2.

4.4.3 Corrective Action for LCSD Precision Failure

- check calculations;
- check instrument performance;
- if the RPD is out of control but both accuracy recoveries are within acceptance criteria, prepare an NCM, and qualify report.

Note: Because LCS/LCSD limits are based on the standard deviation of data collected over time and include long-term precision, it would be unusual to fail precision limits while meeting accuracy limits. If this occurs with any frequency, control limits should be reevaluated.

4.5 SURROGATE EVALUATION (ALSO SEE FIGURE 3)

4.5.1 Acceptance Criteria

Surrogate recoveries must be within established control limits. Method QC (MB, LCS, and/or LCSD) results are not acceptable unless the surrogate recoveries for those QC samples are within control limits. If MS/MSD, DU or field samples require dilutions beyond the threshold stated in the analytical SOPs, routine surrogate control limits do not apply and recoveries are not evaluated. This should be noted in the final report. The recovery is calculated as follows:

$$\textbf{\textit{Surrogate Percent Recovery}} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of surrogate added

4.5.2 Corrective Action

4.5.2.1 Surrogate Failures in MB, LCS, or LCSD

- check calculation and instrument performance,
- reanalyze QC sample and/or reanalyze all samples in the QC batch.

Note: Unless otherwise specified by the client, it may be possible to report qualified results if method QC surrogate recoveries are biased high and analytes were not detected in the field samples. However, all other QC requirements would have to be met and the failure would have to be noted in the final report.

4.5.2.2 Surrogate Failures in Field Samples or MS/MSD

- check calculation and instrument performance;
- evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses);
- document the failure and note it on the final report;

Note: Some client programs require reanalysis to confirm matrix interferences. Check special project instructions for this corrective action.

4.6 MATRIX SPIKE AND MATRIX SPIKE DUPLICATES (MS/MSD) EVALUATION (ALSO SEE FIGURE 4)

4.6.1 Acceptance Criteria

MS and MSD recoveries and RPD should be within established control limits.

If MS or MSD samples require dilutions beyond the threshold stated in the analytical SOPs, routine control limits do not apply and recoveries are not evaluated, but this should be noted in the final report. The MS and MSD recoveries are calculated as follows:

$$\textbf{\textit{MS or MSD Percent Recovery}} = \left[\frac{X_s - X}{t}\right] \times 100$$

Where: X = observed concentration in unspiked sample

X_s = observed concentration in spiked sample

t = concentration of spike added

Notes: 1. If sample result is ND, $X = 0$ when no values reported below RL.

If sample result is reported as a value <RL, X = reported value.

2. CLP forms software uses observed recovery, not concentrations.

RPD is defined in Section 4.4.1.

4.6.2 Corrective Action for MS/MSD or MS/MSD RPD Failure (assuming that the LCS is in control)

- check calculation and instrument performance,
- consider objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses);

- document the failure and note on final report;

  Note: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

4.7 Sample Duplicate (DU)

4.7.1 Acceptance Criteria
The RPD for the sample and its duplicate must be within established control limits. The RPD is the same as for the MS/MSD (see 4.6.1 above).

4.7.2 Corrective Action for DU Failure
- check calculation and instrument performance,
- document the QC failure and note on the final report.

5.0 ESTABLISHING QC ACCEPTANCE LIMITS

5.1 Selecting the Data Set
For new procedures, published method limits can be used until sufficient QC data are acquired (minimum of 20 to 30 data points recommended). However, the published limits may not be appropriate if they are based on a single-operator or single-laboratory study. In this case, the QA Manager may establish default limits until enough data is collected for laboratory established limits to be determined. For existing procedures, data collected over several months to a year or more can be used. Control tables or control charts are used together with the calculated mean and standard deviation to determine if the data set being considered are free of trends and are representative. If it appears that the data include gross outliers, outlier tests such as the Grubbs Test, Dixon Test (for 20 or fewer data points), or Rule-of-Huge-Error Test can be used to justify eliminating individual data points. Laboratory established limits must be reevaluated at least annually.

5.2 CALCULATING LABORATORY STATISTICAL PERFORMANCE

Accuracy: mean recovery $\pm 3s$
Precision: zero to (mean RPD + 3s)

Where: s = standard deviation

If there are insufficient MS/MSD data available to calculate limits, LCS/LCSD limits may be used. If there are insufficient sample surrogate recovery data available to calculate limits, method limits may be used, if available. For methods, matrices, and/or

analytes with very limited data, interim limits should be established using available data or by analogy to similar methods or matrices.

5.3 Setting Control Limits

The working control limits to be used by the laboratory are based on evaluation of the calculated laboratory statistical performance and available interlaboratory limits provided in the reference methods. Note that some SW-846 methods only supply single-operator or single-laboratory method performance data, which should be used for guidance only and may not be appropriate for routine lab operation.

The control limits should be evaluated for systematic trends and consistency of the performance of the analytical procedure at least annually or whenever new patterns of performance are observed in the laboratory data (i.e. new methods, equipment, etc.). When evaluating current "laboratory-generated limits" against historical "laboratory-generated limits", the laboratory QA manager should investigate any significant changes in "laboratory-generated mean" and "laboratory-generated range" and should attempt to identify the cause before making any changes to the laboratory limits. If the recalculated limits are consistent with the historical limits, the historical limits may remain unchanged.

Notes: Laboratory-generated mean = statistical mean (i.e. $\Sigma x_i / n$)

Laboratory-generated range = statistical ranges indicated in the previous section

2. If the laboratory-generated mean is within ± 10% of the historical mean, the two means are not significantly different and no investigation of cause of change would be necessary.
3. If the in-house calculated data lead to limits that are significantly tighter than both the guidance limits and/or the calibration acceptance criteria for the method, the laboratory can default to using the laboratory-generated mean ± calibration acceptance limit. Unreasonably tight statistical limits can result from the exclusion of unacceptable results from the database. If investigation demonstrates that this is happening, the laboratory's data entry systems should be improved. The lab should employ a statistical test for outlier values before excluding data points. (Reiterations of outlier tests lead to narrower limits and should be used with caution.)
4. If the decision is to use guidance limits from the method, the laboratory should investigate procedural improvements leading to better performance.

6.0 REPORTING QC DATA

The QC data routinely reported includes the LCS, method blank and surrogate standards. Matrix QC are reported on a project or client basis, and clients are encouraged to identify on the custody forms specific samples to be used for matrix spiking. Client reporting requirements are negotiated and documented as part of the project records. Ultimately, all reporting decisions should accommodate the client's requirements.

Figure 1 - Method Blank Evaluation

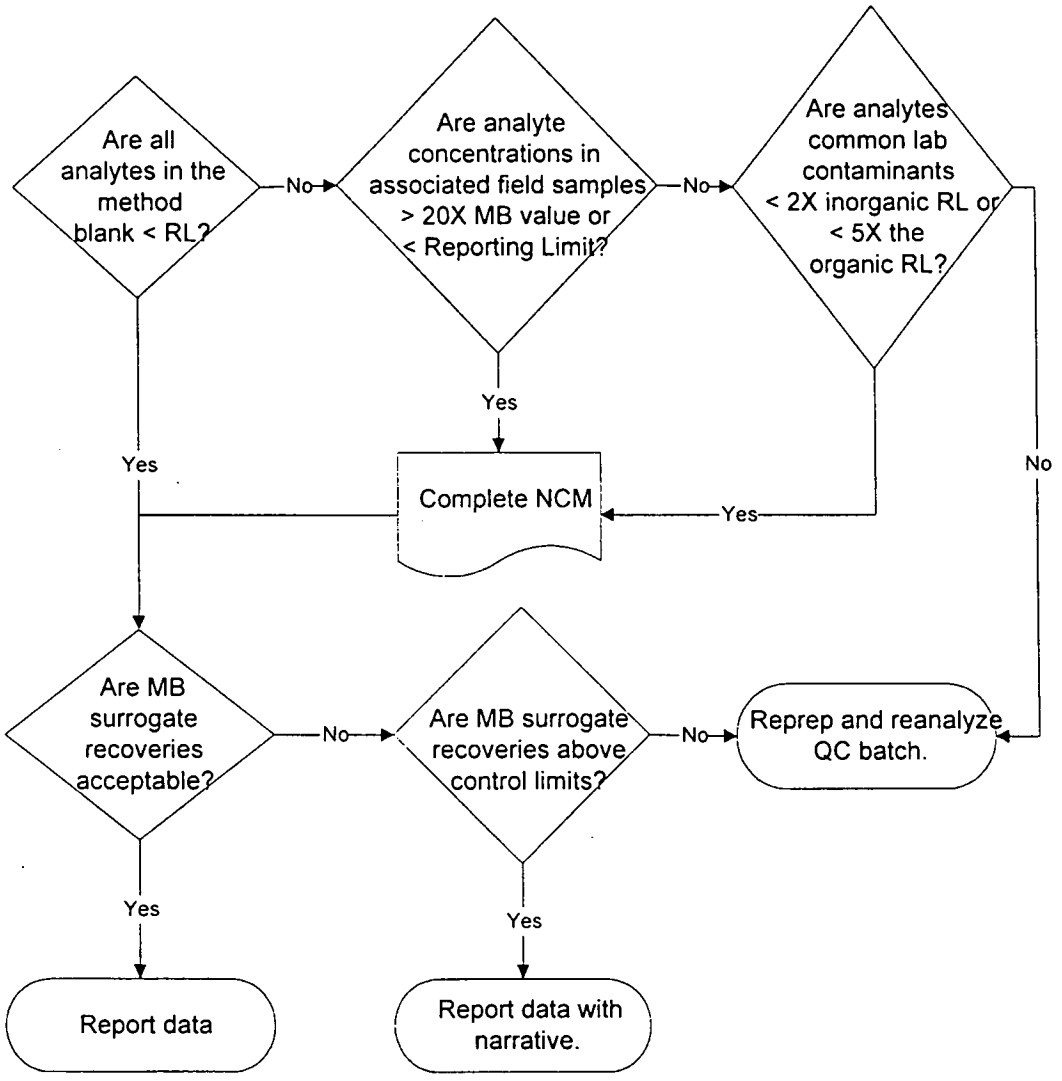
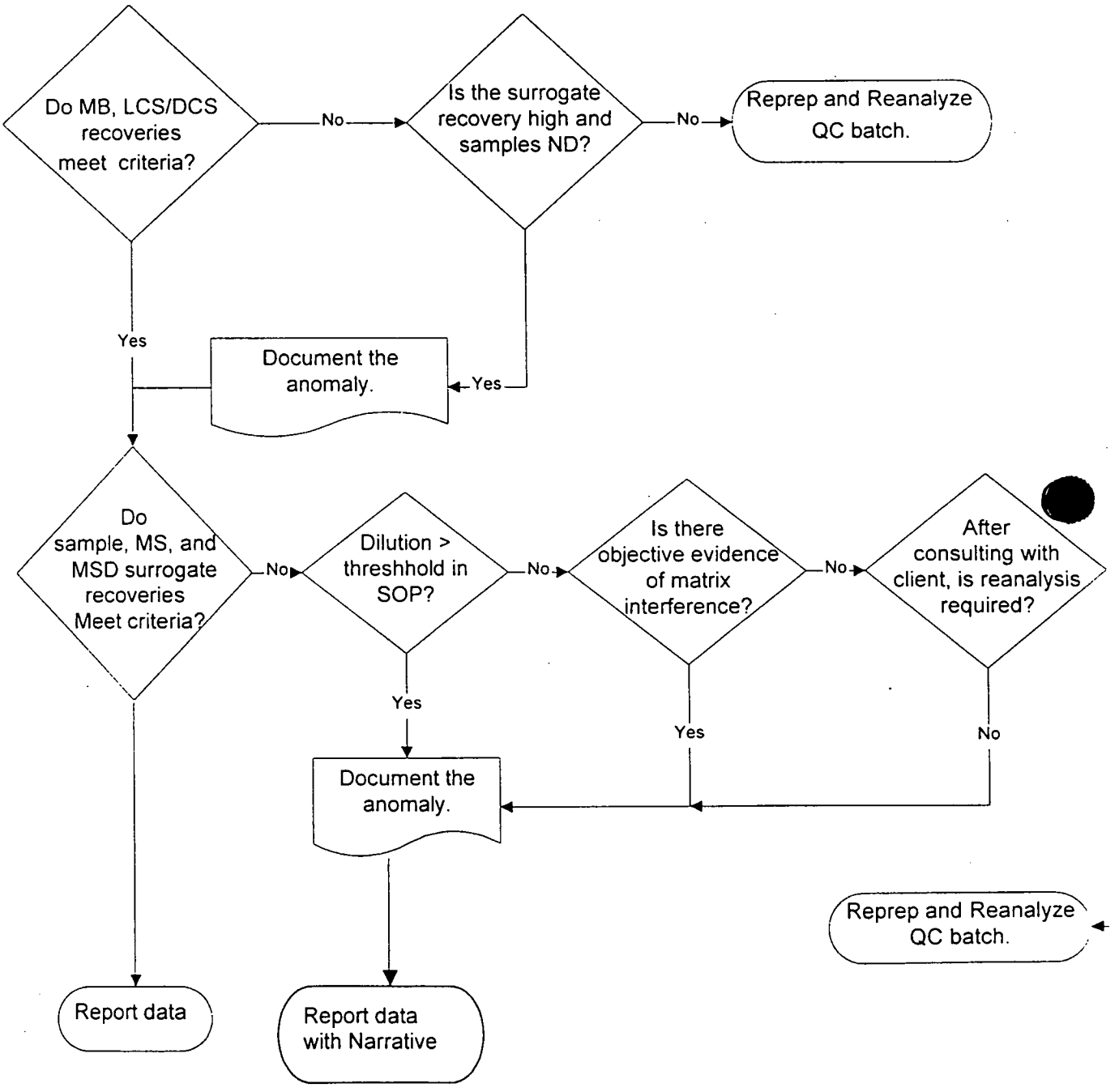
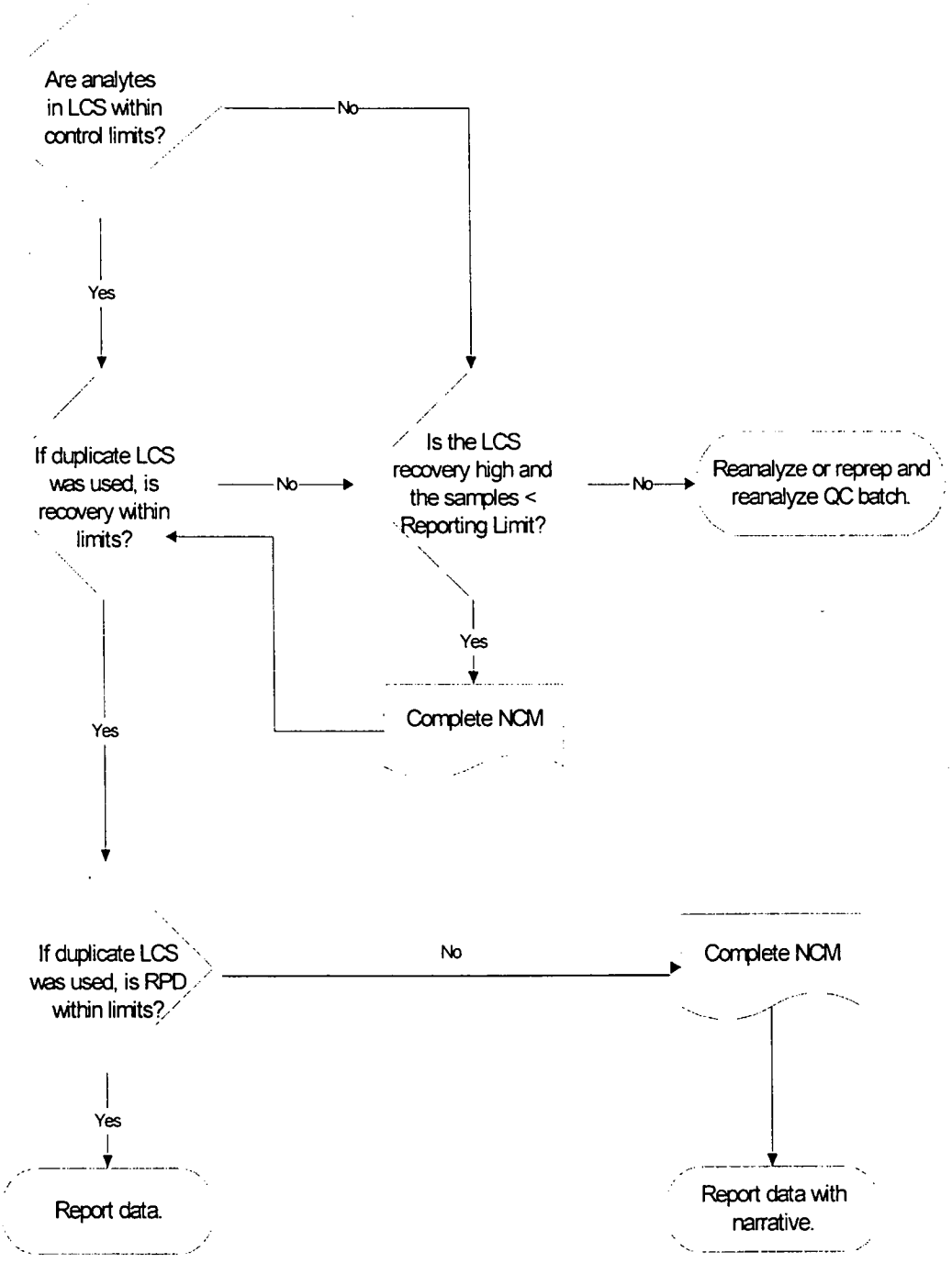
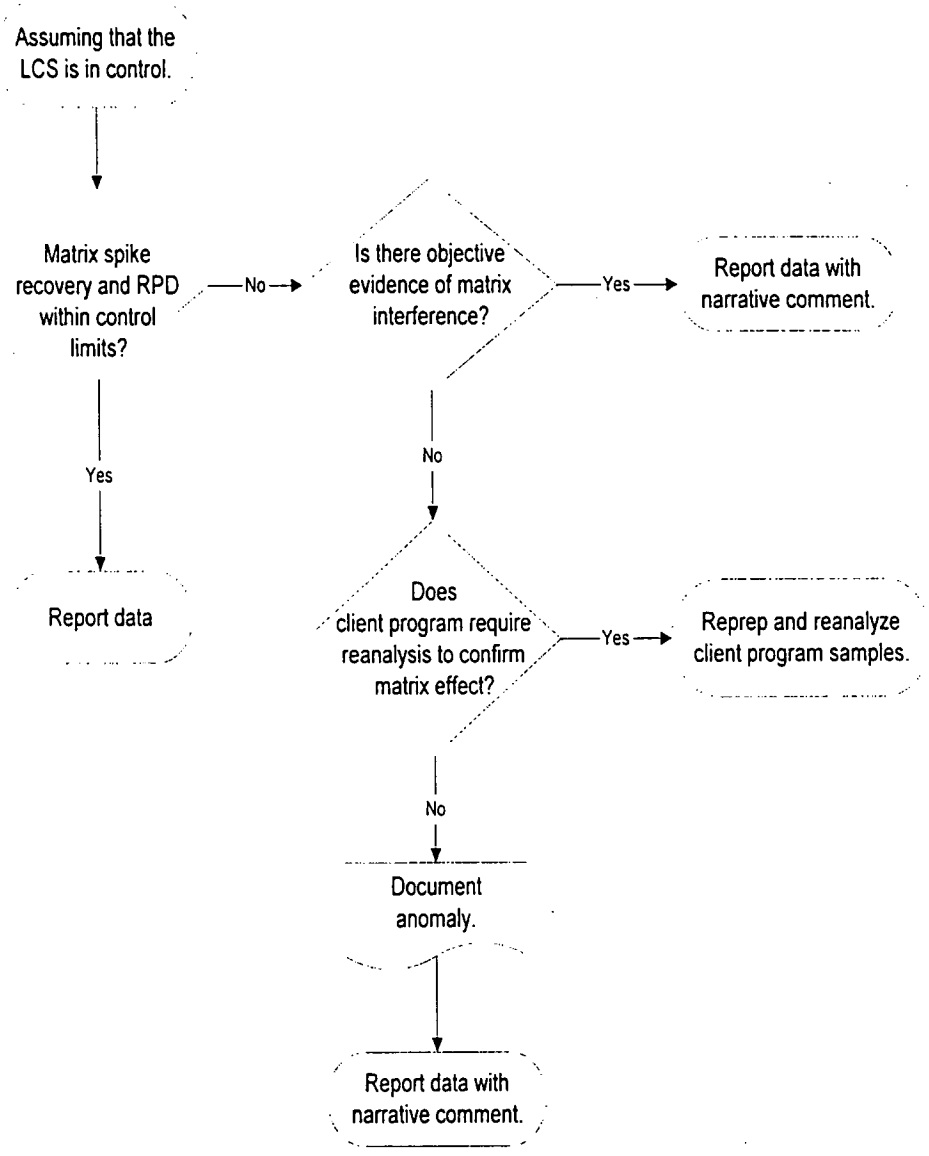


Figure 2 - LCS/LCSD Evaluation

Figure 3 - Surrogate Evaluation




Are analytes in LCS within control limits?
No
Yes
If duplicate LCS was used, is recovery within limits?
No
Is the LCS recovery high and the samples < Reporting Limit?
No
Reanalyze or reprep and reanalyze QC batch.
Yes
Complete NCM
Yes
If duplicate LCS was used, is RPD within limits?
No
Complete NCM
Yes
Report data.
Report data with narrative.


Assuming that the LCS is in control.
Matrix spike recovery and RPD within control limits?
No
Is there objective evidence of matrix interference?
Yes
Report data with narrative comment.
Yes
Report data
No
Does client program require reanalysis to confirm matrix effect?
Yes
Reprep and reanalyze client program samples.
No
Document anomaly.
Report data with narrative comment.

ATTACHMENT 2
QUANTERRA® RADIOCHEMISTRY QUALITY CONTROL PROGRAM

1.0 INTRODUCTION

This Attachment describes the general quality control (QC) program in place to support radiochemical analyses performed in Quanterra® laboratories. Due to the lack of a national QC program which has been deemed to be the industry standard for radiochemistry, individual procedures may vary in their requirements. The program defined here should be taken as the minimum QC program. The details of the implementation and acceptability of the program are described in laboratory SOPs. The requirements of this Attachment may be superseded by a project Scope of work or QA program.

All data produced by the Quanterra® radiochemistry laboratories shall be reviewed by procedures described in the Quanterra® QAMP and facility SOPs. The review will be documented using a data review check list.

2.0 QUALITY CONTROL SAMPLES

2.1 Sample batches (defined in Attachment 1 of this policy) will be accompanied by laboratory prepared QC samples. The minimum QC samples required in a batch are as follows:

- 2.1.1 Method Blanks: carried through the entire preparation and analysis procedure, one for each batch.
- 2.1.2 Laboratory Control Samples: prepared and/or analyzed with the samples, one for each batch.
- 2.1.3 Sample Duplicates: one for each batch, assuming sufficient sample is received.

2.2 Additional Quality Control Samples may include matrix spikes and matrix spike duplicates (matrix spike duplicates will replace sample duplicates) which are performed when contractually required. Matrix spikes are not prepared for samples which are counted directly and are subject to homogenization restrictions, such as solid samples for gamma or alpha/beta analysis.

2.3 Tracers and carriers are added to every sample, blank, and QC sample when required by the analytical method. Tracers are radioisotopes usually with the same atomic number as the target analytes, when available. Carriers are non-radioactive compounds added to assist in isolation of the target analyte(s). Both tracers and carriers, when standardized can be used to correct counting results for actual recovery of the analyte when available radioactive tracers are NIST traceable.

3.0 ACCEPTABILITY CRITERIA/CORRECTIVE ACTION

3.1 In the absence of specific client instructions, the following criteria apply. However, individual cases allow for exceptions as stated below:

3.1.1 Re-counts - Client and/or QC samples are recounted if the acceptance criteria have not been met as indicated on the Data Review Checklist and as deemed necessary by the team leader/designee. Additionally, any blank, spike or client sample may be recounted to verify a result. If the re-count result is found to be within limits, the Data Reviewer accepts the re-count data. If the re-count result is found not to be within limits, the affected samples may need to be reanalyzed.

3.1.2 Reanalysis - If sample reanalysis is necessary and/or the client provides permission, the affected samples and associated quality control samples shall be reanalyzed providing sufficient sample is available. The data of the reanalysis set shall be reviewed using the same criteria as the original data set.

3.2 Method Blank: In general, method blanks may not contain concentrations of the analyte(s) of interest above the Reporting Limit (RL) or Client-Required Detection Limit (CRDL). Specific client requirements may require other criteria for the method blank. The occurrence of an unacceptable method blank requires investigation into the source of the contamination, followed by reanalysis of the associated batch of samples with an acceptable method blank unless the activity levels of the samples are significantly higher than the blank and or the MDA or MDC. The method blank may be water for all matrices unless client requirements specify otherwise

3.3 Yield Monitors: - For QC and sample data where gravimetric (chemical) or tracer recoveries are used for yield correction, the yield of the sample must meet, in the absence of specific limits, a yield monitor recovery greater than or equal to 20% and less than or equal to 115%. Unacceptable yields for method blanks or LCS require investigation and reanalysis of the associated sample batch with acceptable QC sample yields. Unacceptable yields for a sample require reanalysis of the sample in a batch with acceptable QC sample yields. Samples may be reported

with unacceptable yield only if repeat analyses indicate that a matrix effect exists that prevents acceptable yield.

3.4 Laboratory Control Sample: Criteria for acceptability of Laboratory Control Samples are set by each procedure, and may utilize historical LCS recovery data. The occurrence of an unacceptable LCS requires investigation into the source of the nonconformance, followed by reanalysis of the associated batch of samples with an acceptable LCS.

3.5 Duplicate Samples: Acceptability criteria for duplicate sample results are stated in laboratory Standard Operating Procedures, and may utilize historical duplicate RPD data. Data associated with duplicate analyses that exceed the RPD criteria are evaluated for usability, and if reported without corrective action, are flagged and noted in client reports.

3.6 Matrix Spike Recoveries: Acceptable matrix spike recovery is determined from laboratory historical data. Control limits are established for acceptability for each method and matrix at least annually. Samples with unacceptable matrix spike recoveries are flagged and noted in client reports unless the recovery can be shown to be due to laboratory error.

4.0 MDAs/MDCs

4.1 All radiochemical results are reported with both a sample-specific Minimum Detectable Activity (MDA) or Minimum Detectable Concentration (MDC) and an associated error. Formulas for calculating the MDA or MDC and the error are given in the Standard Operating Procedures. If the sample MDA/MDC is greater than the RL or CRDL, the Data Reviewer shall examine the sample volume/weight, counting time, and detector efficiency. The Data Reviewer shall decide if the sample should be recounted, reanalyzed, or the data accepted. If the sample activity exceeds the achieved MDA/MDC, no further action is necessary.

ATTACHMENT D

REMEDIAL GOALS

Attachment D
Battery Area QAPP

All results in mg/kg		SOIL SCREENING LEVEL				
	Analytical		EPA R9 PRGs		Ecological	Lab Reporting
CONSTITUENT	Method	Ingestion	Residential	Industrial	DQL Soil	Limit
SOIL AREA SAMPLES						
Explosives (5samples)						
Pentaerythritol tetranitrate (PETN)	8330	NA	NA	*	NA	0.5
2,4,6-Trinitrotoluene (TNT)	8330	NA	16	82	NA	0.5
Cyclotrimethylene trinitraamine (RDX)	8330	NA	4.4	22	NA	0.625
Cyclotetremethylene tetranitramine (HMX)	8330	NA	3100	44,000	NA	1.6
Tetryl	8330	NA	610	8,800	NA	0.38
Trinitrobenzene	8330	NA	3.3	34	NA	0.575
1,3-dintrobenzene	8330	NA	6.1	88	NA	0.49
4-amino 2,6-dinitrotoluene	8330	NA	65	680	NA	0.45
2-amino 4,6-Dinitrotoluene	8330	NA	130	1,400	NA	0.4
2,4-DNT	8330	NA	120	1,800	NA	0.25
2,6-DNT	8330	NA	61	880	NA	0.39
2-nitrotoluene	8330	NA	370	1,000	NA	0.72
3 NT	8330	NA	370	1,000	NA	0.5
4NT	8330	NA	370	1,000	NA	0.48
Metals (4 samples)						
Aluminum	6010	NA	76000	100,000	NA	20
Barium	6010	5500	5400	100000	1	20
Cadmium	6010	78	37	810	0.002	0.5
Chromium (total)	6010	390	210	450	0.4	1
Lead	6010	400	400	750	0	10
Mercury	7471	23	23	610	0.1	0.04
Volatile Organics (4 samples)						
Dichloromethane (methylene chloride)	8260	85	8.9	21	4.05	0.01
Acetone	8260	7800	1600	6200	NA	0.01
Methyl ethyl ketone	8260	NA	7300	28000	90	0.01
Methyl isobutyl ketone	8260	NA	790	2900	443	0.01
Toluene	8260	16000	520	520	5.45	0.005
Xylene (total)	8260	160000	210	210	10	0.005
Trans 1,3-dichloropropene	8260	NA	0.25	0.55	0.398	0.005
1,1,1-trichloroethane	8260	NA	630	1400	30	0.005
1,1,2-trichloroethane	8260	11	0.84	1.9	28.6	0.005
Appendix IX Constituents (1 sample)						
Acenaphthene	8270	4700	3700	38000	NA	0.33
Acenaphthylene	8270	NA	NA	NA	NA	0.33
Acetone	8260	7800	1600	6200	NA	0.01
Acetophenone	8270	NA	0.49	1.6	NA	0.33
Acetonitrile	8260	NA	270	1700	NA	0.01
2-Acetylaminofluorene	8270	NA	NA	NA	NA	3.3
Acrolein	8260	NA	0.1	0.34	NA	0.1
Acrylonitrile	8260	NA	0.21	0.51	NA	0.1
Aldrin	8081	0.04	0.029	0.15	NA	0.0017
Allyl chloride	8260	NA	3000	43000	NA	0.005
4-Aminobiphenyl	8270	NA	NA	NA	0.003	1.6
Aniline	8270	NA	85	430	0.057	0.33
Anthracene	8270	23000	22000	100000	1480	0.33
Antimony	6020	31	31	820	0.142	0.2
Aramite	8270	NA	19	99	166	0.66
Arsenic	6020	0.4	0.39	2.7	5.7	0.2
Barium	6020	5500	5400	100000	1.04	0.1
Benzene	8260	22	0.65	1.5	0.255	0.005
Benzo[a]anthracene	8270	0.9	0.62	2.9	5.21	0.33
Benzo[b]fluoranthene	8270	0.9	0.62	2.9	59.8	0.33
Benzo[k]fluoranthene	8270	9	6.2	29	148	0.33
Benzo[g,h,i]perylene	8270	NA	NA	NA	119	0.33
Benzo[a]pyrene	8270	0.09	0.062	0.29	1.52	0.33
Benzyl alcohol	8270	NA	18000	100000	65.8	0.33
Beryllium	6020	0.1	150	2200	1.06	0.1
alpha-BHC	8081	0.1	NA	0.3	0.099	0.0017
beta-BHC	8081	0.4	NA	1.1	0.004	0.0017
delta-BHC	8081	NA	NA	NA	9.94	0.0017
gamma-BHC;Lindane	8081	0.5	NA	1.5	0.005	0.0017
Bis(2-chloroethoxy)methane	8270	NA	NA	NA	0.302	0.33
Bis(2-chloroethyl)ether	8270	0.6	0.21	0.62	NA	0.33
Bis(2-chloro-1-methylethyl)ether	8270	NA	2.9	8.1	NA	0.33
Bis(2-ethylhexyl)phthalate	8270	46	35	180	0.926	0.33
Bromodichloromethane	8260	10	1	2.4	0.54	0.005
Bromoform	8260	81	62	310	15.9	0.005
4-Bromophenyl phenyl ether	8270	NA	NA	NA	NA	0.33
Butyl benzyl phthalate	8270	16000	12000	100000	0.239	0.33
Cadmium	6020	78	37	810	0.002	0.1
Carbon disulfide	8260	7800	360	720	0.094	0.01
Carbon tetrachloride	8260	5	0.24	0.53	2.98	0.005
Chlordane	8081	0.5	1.6	11	0.224	0.0017
p-Chloroaniline	8270	310	240	3500	1.1	0.33
Chlorobenzene	8260	1600	150	540	13.1	0.005
Chlorobenzilate	8270	NA	1.8	9.1	5.05	0.33
p-Chloro-m-cresol	8270	NA	NA	NA	7.95	0.33
Chloroethane	8260	NA	3	6.5	NA	0.005

Attachment D
Battery Area QAPP

All results in mg/kg		SOIL SCREENING LEVEL				
	Analytical		EPA R9 PRGs		Ecological	Lab Reporting
CONSTITUENT	Method	Ingestion	Residential	Industrial	DQL Soil	Limit
Chloroform	8260	100	0.24	0.52	1.19	0.005
2-Chloronaphthalene	8270	NA	3900	27000	0.012	0.33
2-Chlorophenol	8270	390	63	240	0.243	0.33
4-Chlorophenyl phenyl ether	8270	NA	NA	NA	NA	0.33
Chloroprene	8260	NA	3.6	12	0.003	0.01
Chromium (total)	6020	390	210	450	0.4	0.2
Chrysene	8270	88	62	290	4.73	0.33
Cobalt	6020	NA	4700	100000	0.14	0.1
Copper	6020	NA	2900	76000	0.313	0.2
m-Cresol	8270	NA	3300	34000	3.49	0.33
o-Cresol	8270	3900	NA	34000	40.4	0.33
p-Cresol	8270	NA	330	3400	163	0.33
Cyanide	9012	1600	NA	14000	1.33	0.5
2,4-D	8151	NA	690	12000	0.027	0.08
4,4'-DDD	8081	3	2.4	17	0.758	0.0034
4,4'-DDE	8081	2	1.7	12	0.596	0.0034
4,4'DDT	8081	2	1.7	12	0.018	0.0034
Diallate	8270	NA	8	40	0.452	0.66
Dibenz[a,h]anthracene	8270	0.09	0.062	0.29	18.4	0.33
Dibromochloromethane	8260	8	1.1	2.7	2.05	0.005
1,2-Dibromo-3-chloropropane	8260	NA	0.45	4	0.035	0.01
1,2-Dibromoethane; Ethylene	8260	NA	0.0069	0.048	1.23	0.01
Di-n-butyl phthalate	8270	7800	6100	88000	0.15	0.33
o-Dichlorobenzene	8260	7000	370	370	2.96	0.005
m-Dichlorobenzene	8260	NA	13	52	37.7	0.005
p-Dichlorobenzene	8260	27	3.4	8.1	0.546	0.005
3,3-Dichlorobenzidine	8270	1	1.1	5.5	0.646	1.6
trans-1,4-Dichloro-2-butene	8260	NA	NA	NA	NA	0.01
Dichlorodifluoromethane	8260	NA	94	310	39.5	0.005
1,1-Dichloroethane	8260	7800	590	2100	20.1	0.005
1,2-Dichloroethane	8260	7	0.35	0.76	21.2	0.005
1,1-Dichloroethylene	8260	1	0.054	0.12	8.28	0.005
trans-1,2-Dichloroethylene	8260	1600	63	210	0.784	0.005
2,4-Dichlorophenol	8270	230	180	2600	87.5	0.33
2,6-Dichlorophenol	8270	NA	NA	NA	1.17	0.33
1,2-Dichloropropane	8260	9	0.35	0.77	32.7	0.005
1,3-Dichloropropene (mixture)	8260	4	0.7	1.6	NA	0.005
cis-1,3-Dichloropropene	8260	NA	NA	NA	0.398	0.005
trans-1,3-Dichloropropene	8260	NA	0.25	0.55	0.398	0.005
Dieldrin	8081	0.04	0.03	0.15	0.002	0.0034
Diethyl phthalate	8270	63000	49000	100000	24.8	0.33
O,O-Diethyl O-2-pyrazinyl phosphorothioate	8270	NA	NA	NA	0.8	1.6
Dimethoate	8270	NA	12	180	0.218	0.66
p-(Dimethylamino)azobenzene	8270	NA	NA	NAA	0.04	0.66
7,12-Dimethylbenz[a]anthracene	8270	NA	NA	NAA	16.3	0.66
3,3-Dimethylbenzidine	8270	NA	0.053	0.27	0.104	1.6
alpha, alpha-Dimethylphenethylamine	8270	NA	61	880	0.3	1.6
2,4-Dimethylphenol	8270	1600	1200	14000	0.01	0.33
Dimethyl phthalate	8270	NA	100000	100000	734	0.33
m-Dinitrobenzene	8270	NA	6.1	88	0.655	0.33
4,6-Dinitro-o-cresol	8270	NA	NA	NA	0.144	1.6
2,4-Dinitrophenol	8270	160	120	1800	0.061	1.6
2,4-Dinitrotoluene	8270	0.9	120	1800	1.28	0.33
2,6-Dinitrotoluene	8270	0.9	61	880	0.033	0.33
Dinoseb	8270	NA	61	880	0.022	0.66
Di-n-octyl phthalate	8270	1600	1200	10000	NA	0.33
1,4-Dioxane	8260	NA	44	220	2.05	0.25
Diphenylamine	8270	NA	1500	22000	1.01	**
Disulfoton	8270	NA	2.4	35	0.02	1.6
Endosulfan I	8081	NA	NA	NA	0.119	0.0017
Endosulfan II	8081	NA	NA	NA	0.119	0.0034
Endosulfan sulfate	8081	NA	NA	NA	0.036	0.0034
Endrin	8081	23	18	260	0.01	0.0034
Endrin aldehyde	8081	NA	NA	NA	0.011	0.0034
Ethylbenzene	8260	7800	230	230	5.16	0.005
Ethyl methacrylate	8260	NA	140	140	30	0.01
Ethyl methanesulfonate	8270	NA	NA	NA	NA	0.33
Famphur	8270	NA	NA	NA	0.05	3.3
Fluoranthene	8270	3100	2300	30000	122	0.33
Fluorene	8270	3100	2600	33000	122	0.33
Heptachlor	8081	0.1	0.11	0.55	0.006	0.0017
Heptachlor epoxide	8081	0.07	0.053	0.27	0.152	0.0017
Hexachlorobenzene	8270	0.4	0.3	1.5	0.199	0.33
Hexachlorobutadiene	8270	8	6.2	32	0.04	0.33
Hexachlorocyclopentadiene	8270	550	420	5900	0.755	1.6
Hexachloroethane	8270	46	35	180	0.596	0.33
Hexachlorophene	8270	NA	18	260	0.199	**
Hexachloropropene	8270	NA	NA	NA		3.3
2-Hexanone	8260	NA	NA	NA	12.6	0.01
Indeno(1,2,3-cd)pyrene	8270	0.9	0.62	2.9	109	0.33

Attachment D
Battery Area QAPP

All results in mg/kg		SOIL SCREENING LEVEL				
	Analytical		EPA R9 PRGs		Ecological	Lab Reporting
CONSTITUENT	Method	Ingestion	Residential	Industrial	DQL Soil	Limit
Isobutyl alcohol	8260	NA	13000	40000	20.8	0.1
Isodrin	8270	NA	NA	NA	0.003	0.33
Isophorone	8270	670	510	2600	139	0.33
Isosafrole	8270	NA	NA	NA	9.94	0.66
Kepone	8270	NA	0.027	0.14	0.033	3.3
Lead	6020	400	400	750	0.054	0.1
Mercury (total)	7471	23	23	610	0.1	0.04
Methacrylonitrile	8260	NA	2.1	8.8	0.057	0.01
Methapyrilene	8270	NA	NA	NA	2.78	1.6
Methoxychlor	8081	390	310	4400	0.02	0.017
Methyl Bromide; Bromomethane	8260	110	3.9	13	0.235	0.005
Methyl chloride; Chloromethane	8260	NA	1.2	2.7	10.4	0.005
3-Methylcholanthrene	8270	NA	NA	NA	0.078	0.66
Methylene bromide; Dibromomethane	8260	NA	67	240	0.235	0.005
Methylene chloride; Dichloromethane	8260	85	8.9	21	4.05	0.01
Methyl ethyl ketone; MEK	8260	NA	7300	28000	89.6	0.01
Methyl iodide; Iodomethane	8260	NA	NA	NA	1.23	0.01
Methyl methacrylate	8260	NA	2200	2700	984	0.01
Methyl methanesulfonate	8270	NA	NA	NA	0.316	0.33
2-Methylnaphthalene	8270	NA	NA	NA	3.24	0.33
Methyl parathion	8270	NA	15	220	0.003	1.6
4-Methyl-2-pentanone; Methyl isobutyl ketone	8260	NA	790	2900	443	0.01
Naphthalene	8270	3100	56	190	0.099	0.33
1,4-Naphthoquinone	8270	NA	NA	NA	1.67	1.6
1-Naphthylamine	8270	NA	NA	NA	9.34	0.33
2-Naphthylamine	8270	NA	NA	NA	3.03	0.33
Nickel	6020	1600	1600	41000	13.6	0.2
o-Nitroaniline	8270	NA	3.5	50	74.1	1.6
m-Nitroaniline	8270	NA	3.9	NA	3.16	1.6
p-Nitroaniline	8270	NA	NA	NA	21.9	1.6
Nitrobenzene	8270	39	20	110	1.31	0.33
o-Nitrophenol	8270	NA	NA	NA	1.6	0.33
p-Nitrophenol	8270	NA	490	7000	5.12	1.6
4-Ntiroquinoline 1-oxide	8270	NA	NA	NA	0.122	3.3
N-Nitrosodi-n-butylamine	8270	NA	0.024	0.61	0.267	0.33
N-Nitrosodiethylamine	8270	NA	0.0032	0.016	0.069	0.33
N-Nitrosodimethylamine	8270	NA	0.0095	0.048	0.003	0.33
N-Nitrosodiphenylamine	8270	130	99	500	0.545	0.33
N-Nitrosodipropylamine	8270	0.09	0.069	0.35	NA	0.33
N-Nitrosomethylethylamine	8270	NA	0.022	0.11	0.002	0.33
N-Nitrosomorpholine	8270	NA	NA	NA	0.071	0.33
N-Nitrosopiperidine	8270	NA	NA	NA	0.007	0.33
N-Nitrosopyrrolidine	8270	NA	0.23	1.2	0.013	0.33
5-Nitro-o-toluidine	8270	NA	13	58	8.73	0.66
Parathion	8270	NA	370	5300	0.003	1.6
PCBs	8082	1	0.22	1	0.003	0.033
Pentachlorobenzene	8270	NA	4.9	700	0.497	0.33
Pentachloroethane	8270	NA	NA	NA	10.7	1.6
Pentachloronitrobenzene	8270	NA	1.9	9.5	7.09	1.6
Pentachlorophenol	8270	3	3	11	0.119	1.6
Phenacetin	8270	NA	NA	NA	11.7	0.66
Phenanthrene	8270	NA	100	NA	45.7	0.33
Phenol	8270	47000	37000	100000	120	0.33
p-Phenylenediamine	8270	NA	12000	100000	6.16	3.3
Phorate	8270	NA	12	180	0.005	1.6
2-Picoline	8270	NA	NA	NA	9.9	0.66
Pronamide	8270	NA	4900	51000	0.014	0.66
Propionitrile; Ethyl Cyanide	8260	NA	NA	NA	0.05	0.01
Pyrene	8270	2300	2300	54000	78.5	0.33
Pyridine	8270	NA	61	880	1.03	0.66
Safrole	8270	NA	NA	NA	0.404	0.66
Selenium	6020	390	390	10000	0.028	0.2
Silver	6020	390	390	10000	4.04	0.1
Silvex; 2,4,5-TP	8151	NA	490	7000	0.109	0.02
Styrene	8260	16000	1700	1700	4.69	0.005
Sulfide	9030	NA	NA	NA	0.004	50
2,4,5-Trichlorophenoxyacetic acid	8151	NA	610	8800	0.596	0.02
1,2,4,5-Tetrachlorobenzene	8270	NA	18	260	2.02	0.33
1,1,1,2-Tetrachloroethane	8260	NA	3	7	225	0.005
1,1,2,2-Tetrachloroethane	8260	3	0.38	0.9	0.127	0.005
Tetrachloroethylene	8260	12	5.7	19	9.92	0.005
2,3,4,6-Tetrachlorophenol	8270	NA	1800	26000	0.199	1.6
Tetraethyl dithiopyrophosphate	8270	NA	31	440	0.596	1.6
Thallium	6020	NA	5.4	120	0.057	0.1
Tin	6020	NA	46000	100000	7.62	1
Toluene	8260	16000	520	520	5.45	0.005
o-Toluidine	8270	NA	NA	NA	2.97	0.66
Toxaphene	8081	0.6	0.44	2.2	0.119	0.067
1,2,4-Trichlorobenzene	8260	780	650	3000	11.1	0.005
1,1,1-Trichloroethane	8260	NA	630	1400	29.8	0.005

Attachment D
Battery Area QAPP

All results in mg/kg		SOIL SCREENING LEVEL				
	Analytical		EPA R9 PRGs		Ecological	Lab Reporting
CONSTITUENT	Method	Ingestion	Residential	Industrial	DQL Soil	Limit
1,1,2-Trichloroethane	8260	11	0.84	1.9	28.6	0.005
Trichloroethylene	8260	58	2.8	6.1	12.4	0.005
Trichlorofluoromethane	8260	NA	390	2000	16.4	0.005
2,4,5-Trichlorophenol	8270	7800	6100	88000	14.1	0.33
2,4,6-Trichlorophenol	8270	NA	44	220	9.94	0.33
1,2,3-Trichloropropane	8260	NA	0.0014	0.0031	3.36	0.005
O,O,O-Triethyl phosphorothioate	8270	NA	NA	NA	0.818	1.6
sym-Trinitrobenzene	8270	NA	1800	26000	0.376	1.6
Vanadium	6020	550	550	14000	1.59	1
Vinyl acetate	8260	78000	430	1400	12.7	0.01
Vinyl chloride	8260	0.3	0.15	0.83	0.646	0.005
Xylene (total)	8260	160000	210	210	10	0.005
Zinc	6020	23000	23000	100000	6.62	0.5
BATTERY AREA SAMPLES (six samples)						
Ignitability	1010	NA	NA	NA	NA	-
Corrosivity	EPA 150.1	NA	NA	NA	NA	-
Reactivity	Cpt. 7 , SW 846	NA	NA	NA	NA	RS 200; RC 200
Cobalt	6010	NA	4700	100,000	0.14	5
Lithium	6010	NA	1600	41000	NA	5
Manganese	6010	NA	1,800	32,000	NA	1.5
Nickel	6010	1600	1600	41,000	13.6	4
Zinc	6010	23000	23000	100,000	6.62	2
Arsenic	6010 trace	0.4	0.39	2.7	5.7	1
Barium	6010	5500	5400	100000	1.04	20
Cadmium	6010	78	37	810	0.002	0.5
Chromium	6010	390	210	450	0.4	1
Lead	6010	400	400	750	0.054	10
Mercury	7471	23	23	510	0.1	0.04
Selenium	6010	390	390	10,000	0.028	25
Silver	6010	390	390	10,000	4	1

* to be determined

** contract laboratory can not perform this analysis

NA not applicable to this consituent or not available

EPA Region 9 PRGs updated 1/24/01

APPENDIX D

ACTIVITY HAZARD ANALYSIS

ACTIVITY HAZARD ANALYSIS

ACTIVITY Site Preparation/Layout ANALYZED BY/DATE K. Helman 10/98 REVIEWED BY/DATE_____

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS	EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
Site walk through Identification of work zones for construction activities	1.Exposure to irritant and toxic plants such as poison ivy and sticker bushes may cause allergic reactions. 2. Surfaces covered with heavy vegetation and under growth create a tripping hazard. 3. Back strain due to carrying instruments. 4. Native wildlife such as rodents, ticks, and snakes present the possibility of insect bites and associated diseases such as Lyme disease. 5. Driving vehicles on uneven or unsafe surfaces can result in accidents such as overturned vehicles or flat tires. 6. Electrical hazard due to fallen lines. 7. Thermal stress due to hot/cold temperature extremes.	1. Wear long sleeved clothing and slacks to minimize contact with irritant and toxic plants and to protect against insect bites. Appropriate first aid for personnel's known allergic reactions. 2. Be alert and observe terrain while walking to minimize slips and falls. Steel-toed boots provide additional support and stability. 3. Use proper lifting techniques to prevent back strain. 4. Avoid wildlife when possible. In case of an animal bite, perform first aid and capture the animal, if possible, for rabies testing. Perform a tick check after leaving a wooded or vegetated area. 5. Ensure all maintenance is performed on vehicles before going to the field. Site surveillance on foot might be required to choose clear driving paths. 6. Ensure fallen power lines are not energized. 7. Implement thermal stress management techniques such as shifting work hours, fluid intake, and monitoring employees, especially high risk			1. Review hazard analysis with personnel performing the site walk through prior to start

ACTIVITY HAZARD ANALYSIS

ACTIVITY Soil Excavation ANALYZED BY/DATE K. Helman 10/98 REVIEWED BY/DATE _____

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS	EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
Excavation	1. Exposure to airborne contaminants released during intrusive activities. 2. Sides of excavation can cave in. Possible burying or crushing of workers due to 1) absence of shoring, 2) misjudgment of stability, 3) defective shoring, and/or 4) undercut sides. 3. Falling during access/egress or while monitoring or dismounting equipment, or stumbling into excavation. 4. Congested work area due to too many workers in a small area. 5. Existing utilities.	1. Monitor for airborne contaminants. 2. Regularly inspect trenches for conditions. 3.Provide adequate shoring or sloping of sides of the excavation Solid rock, cemented sand or gravel = 90 degrees Compact angular gravel = 63 degrees 26 ft. Compacted sharp sand = 33 degrees 41 ft. deep Rounded loose sand = 26 degrees 34 ft. deep 4. Provide an adequate barrier around open pits. Material from pit must be placed away from edge to prevent cave ins and instability of pit. 5. To prevent overexertion, limit manual lifting and emphasize mechanical means where practical. 6. Maintain ample workroom between workers. 7.Find and mark utilities before excavating utilizing the Joint Utility Locating Information for Excavators (JULIE) service 72 hours prior to excavation activities. Use care while excavating, shore existing utilities crossing excavation area. Watch for overhead lines. 8. Check the performance of JULIE locate prior to digging.	1. Backhoe 2. Rolloff Boxes	Daily, prior to use per manufactures recommendations, Fill our Safety Inspection Checklist Use of assistive climbing equipment when covering or placing tarp on box	OSHA 1910.120 40-hr. training, 3 day OJT, 8 hr. Supervisory, 8 hr. refresher, OSHA Hazard Communication, Respirator and operator training

ACTIVITY HAZARD ANALYSIS

ACTIVITY Soil Sampling ANALYZED BY/DATE K. Helman 10/98 REVIEWED BY/DATE_____

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS	EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
Surface Soil Sampling	1. Dermal contact with or inhalation of contaminants, potentially in high concentrations in sampling media. 2. Back strain and muscle fatigue due to lifting, shoveling and auguring techniques. 3. Dermal contact with or inhalation of decontamination solutions.	1. To minimize exposure to chemical contaminants, a thorough review of suspected contaminants shall be completed and implementation of an adequate protection program. 2. PPE shall include level D consisting of work uniform, steel toe boots/shoes, hard hat, safety glasses, hearing protection (when levels exceed 85 dbs) and Tyvek suit (may not be needed depending on site conditions). 3. Proper lifting (pre-lift weight assessment, use of legs, multiple personnel) techniques will prevent back strain. Use slow easy motions when shoveling, auguring, and digging to decrease muscle strain. 4. Material Safety Data Sheets for all decontamination solutions shall be included with the Site Health and Safety Plan. 5. First aid equipment shall be available based on MSDS requirements.	Soil sampling tools		OSHA 1910.120 40-hr. training, 3 day OJT, 8 hr. Supervisory, 8 hr. refresher, OSHA Hazard Communication, and Respirator training. Knowledge and training on collection of environmental samples

ACTIVITY HAZARD ANALYSIS

ACTIVITY Backfill & Site Restoration ANALYZED BY/DATE K. Helman 10/98 REVIEWED BY/DATE_____

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS	EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
Backfill excavation Compact backfill Seed area	1. Noise levels exceeding the OSHA PEL of 90 dBA are both a hazard and a hindrance to communication. 2. Carbon monoxide from the heavy equipment. 3. Overhead utility wires, i.e., electrical and telephone, can be hazardous when the dump truck bed is in the upright position. 4. Falling backfill material from dump truck may cause injury. 5. Moving the equipment over uneven terrain may cause the vehicle to roll over or get stuck in a rut or mud. Be aware of hazards associated with moving heavy machinery and other associated injury. 6. High-pressure hydraulic lines and airlines used on heavy equipment are hazardous when they are in ill repair or incorrectly assembled.	1. Ear muffs or earplugs effectively reduce noise levels. 2. Review the contaminants suspected to be on-site and perform air monitoring as required. Shut down equipment and/or divert exhaust fumes. 3. All chains, lines, cables shall be inspected daily for weak spots. 4. Hard hats shall be worn at all times when working around heavy equipment. 5. Secure loose clothing. 6. To avoid contact with any overhead lines, the truck bed shall be lowered prior to moving the truck. Overhead utilities shall be considered "live" until determined otherwise. 7. The truck bed should not be erected within 10 feet of an overhead electrical line until the line is de-energized, grounded, or shielded and an electrician has certified that arcing cannot occur. 8. All high-pressure lines shall be checked prior to and during use.	1. Backhoe 2. Compactor	Daily, prior to use per manufactures recommendations, Fill our Safety Inspection Checklist	OSHA 1910.120 40-hr. training, 3 day OJT, 8 hr. Supervisory, 8 hr. refresher, OSHA Hazard Communication, Respirator and operator training OSHA 1910.120 40-hr. training, 3 day OJT, 8 hr. Supervisory, 8 hr. refresher, OSHA Hazard Communication, Respirator and operator training

ACTIVITY HAZARD ANALYSIS

ACTIVITY Soil Loadout REVIEWED BY/DATE K. Helman 10/98 REVIEWED BY / DATE_____

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS	EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
Loadout Soil	1. Noise levels exceeding 85 dbs are both a hazard and a hindrance to communication. 2. Carbon monoxide from the heavy equipment. 3. Overhead utilities. 4. Falling backfill material from backhoe may cause injury. 5. Moving the equipment over uneven terrain may cause the vehicle to roll over or get stuck 6. High-pressure hydraulic lines and airlines used on heavy equipment are hazardous when they are in ill repair or incorrectly assembled.	1. Ear muffs or earplugs effectively reduce noise levels 2. Review the contaminants suspected to be on-site and perform air monitoring as required. Shut down equipment and/or divert exhaust fumes. 3. All chains, lines, cables should be inspected daily for weak spots. 4. Hard hats should be worn at all times when working around a heavy equipment. 5. Secure loose clothing. 6. Overhead utilities should be considered "live" until determined otherwise. 7. Be aware of hazards associated with moving heavy equipment. 8. All high-pressure lines should be checked prior to and during use.	1. Backhoe	Daily, prior to use per manufactures recommendations, Fill our Safety Inspection Checklist	OSHA 1910.120 40-hr. training, 3 day OJT, 8 hr. Supervisory, 8 hr. refresher, OSHA Hazard Communication, Respirator and operator training

ACTIVITY HAZARD ANALYSIS

ACTIVITY Equipment Decontamination ANALYZED BY/DATE K. Helman 10/98 REVIEWED BY/DATE _____

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS	EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
Decontaminate Heavy Equipment Using High Pressure Wash or Hand Scrubbing Decontaminate Sampling Equipment by Hand Washing	1. Contact with contaminated material, inhalation of airborne aerosols, contact with high-pressure wash stream, unexpected movement of material to be decontaminated.	1. Decontamination area to provide isolation and controlled access. 2. Level D PPE with face shield. Secure items to be decontaminated. Visual inspect integrity of containment liners and containers used for wastewater. CRZ provided for worker decontamination. 3. MSDS' obtained and reviewed for all cleaning solutions chemicals if used.	High pressure wash with soap solution, other decontamination solutions, scrub brushes.	Use general safety rules and procedures listed in SSHP, review manufacturer's recommendation and guidance on inspection equipment. Complete on daily basis after use.	OSHA 1910.120 40-Hour Training, 3 day OJT, 8 hours Supervisory, 8 hour Refresher, SSHP, OSHA Hazard Communication and Respirator training.