

**QUALITY ASSURANCE PROJECT PLAN
FOR THE LIMITED SITE INVESTIGATION
PENTOLITE AREA WASTE LAGOONS
@
PLUM BROOK ORDNANCE WORKS**

MARCH 2001

**Prepared by:
U.S. ARMY CORPS OF ENGINEERS
LOUISVILLE DISTRICT**

200-1e
01.09_0004
G050H001819_01.09_0004_a

TABLE OF CONTENTS

TITLE PAGE	
TABLE OF CONTENTS.....	i
<i>List of Acronyms</i>	v
SECTION I	
PROJECT DESCRIPTION.....	1
1.1 Introduction	1
1.2 PBOW Installation History.....	1
1.3 PAWL Site Description.....	2
1.3.1 General	2
1.3.2 PAWL, Site Specific Operational History.....	2
1.3.3 Facility/Size and Border	4
1.3.4 Climate	4
1.3.5 Topography.....	4
1.3.6 Geology	4
1.3.7 Hydrogeology	5
1.4 Past Data Collection Activities and Current Status.....	6
1.5 Project Objectives.....	6
1.5.1 Specific Objectives and Associated Tasks	7
1.5.2 Project Target Parameters and Intended Data Usages.....	7
1.5.3 Quality Objectives and Criteria for Measurement Data	8
1.6 Sample Network Design and Rationale.....	8
1.6.1 Sample Network by Task and Matrix.....	8
1.6.2 Site Maps of Sampling Locations	8
1.6.3 Rationale of Selected Sampling Locations.....	8
1.7 Project Schedule	8
SECTION 2	
PROJECT ORGANIZATION AND RESPONSIBILITY	9
2.1 Project Organization.....	9
2.2 Management Responsibilities.....	10
2.3 Quality Assurance (QA) responsibilities.....	11
2.4 Field Responsibilities	11
2.5 Laboratory Responsibilities.....	12
SECTION 3	
QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA.....	15
3.1 Precision	15
3.1.1 Definition	15
3.1.2 Field Precision Objectives.....	15

3.1.3	Laboratory Precision Objectives	15
3.2	Accuracy.....	15
3.2.1	Definition	15
3.2.2	Field Accuracy Objectives.....	15
3.2.3	Laboratory Accuracy Objectives	15
3.3	Completeness	16
3.3.1	Definition	16
3.3.2	Field Completeness Objectives	16
3.3.3	Laboratory Completeness Objectives	16
3.4	Representativeness	16
3.4.1	Definition	16
3.4.2	Measures to Ensure Representativeness of Field Data.....	16
3.4.3	Measures to Ensure Representativeness of Laboratory Data	16
3.5	Comparability.....	16
3.5.1	Definition	16
3.5.2	Measures to Ensure Comparability of Field Data	17
3.5.3	Measures to Ensure Comparability of Laboratory Data	17
3.6	Level of Quality Control Effort.....	17
 SECTION 4		
SAMPLING PROCEDURES.....		19
 SECTION 5		
CUSTODY PROCEDURES.....		21
5.1	Field Custody Procedures.....	21
5.2	Laboratory Custody Procedures	22
5.3	Final Evidence Files	23
 SECTION 6		
CALIBRATION PROCEDURES AND FREQUENCY.....		24
6.1	Field Instrument Calibration	24
6.2	Laboratory Instrument Calibration	24
 SECTION 7		
ANALYTICAL PROCEDURES.....		26
7.1	Field Analytical Procedures	26
7.2	Laboratory Analytical Procedures	26
7.3	List of Project Target Compounds and Laboratory Detection Limits	26
 SECTION 8		
INTERNAL QUALITY CONTROL CHECKS.....		27
8.1	Field Quality Control Checks.....	27
8.2	Laboratory Quality Control Checks.....	27

SECTION 9	
DATA REDUCTION, VALIDATION, AND REPORTING	28
9.1 Data Reduction	28
9.1.1 Field Data Reduction Procedures	28
9.1.2 Laboratory Data Reduction Procedures	28
9.2 Data Validation.....	29
9.2.1 Procedures Used to Evaluate Field Data	29
9.2.2 Procedures to Validate Laboratory Data.....	29
9.3 Data Reporting	30
9.3.1 Field Data Reporting	30
9.3.2 Laboratory Data Reporting.....	30
SECTION 10	
PERFORMANCE AND SYSTEM AUDITS	32
10.1 Laboratory Performance and Systems Audits	32
10.1.1 Internal laboratory Audits.....	32
10.1.1.1 Internal Lab Audit Responsibilities.....	32
10.1.1.2 Internal Lab Audit Frequency.....	32
10.1.1.3 Internal Lab Audit Process	32
10.1.2 External Laboratory Audits	33
10.1.2.1 External Lab Audit Responsibilities.....	33
10.1.2.2 External Lab Audit Frequency	33
10.1.2.3 Overview of the External Lab Audit Process	33
SECTION 11	
PREVENTATIVE MAINTENANCE.....	34
11.1 Field Instrument Preventive Maintenance	34
11.2 Laboratory Instrument Preventive Maintenance	34
SECTION 12	
SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS	35
12.1 Accuracy Assessment.....	35
12.2 Precision Assessment	35
12.3 Completeness Assessment.....	35
SECTION 13	
CORRECTIVE ACTION.....	37
13.1 Field Corrective Action	37
13.2 Laboratory Corrective Action.....	39
13.3 Corrective Action During Data Validation and Data Assessment	40

SECTION 14

QUALITY ASSURANCE REPORTS TO MANAGEMENT 42
14.1 Contents of Project QA Reports 42
14.2 Frequency of QA Reports 42
14.3 Individuals Receiving/Reviewing QA Reports..... 42

REFERENCES

FIGURES

- Figure 1A: PBOW Site Location Plan
- Figure 1B: PAWL Site Location Plan
- Figure 2: Boring Location Plan
- Figure 3A: Layout Plan of Pentolite Waste Water Basins
- Figure 3B: Construction Details of Pentolite Waste Water Basins

APPENDICES

- A. Ethics Agreements
- B. Quanterra Standard Operating Procedures (SOPs)
- C. Laboratory Analysis Criteria
- D. Data Validation Checklist
- E. Quanterra Reference Data Summary (Method Detection Limits (MDLs) and Method Recovery Limits (MRL) Tables)
- F. Comments and Responses

ATTACHED DOCUMENTS

- I. Field Sampling Plan (FSP)
- II. Data Quality Objectives (DQOs)

ASSOCIATED DOCUMENTS NOT ATTACHED

- I. Site Specific Health and Safety Plan dated May 1999

List of Acronyms

ASTM	American Standards for Testing Materials
BTEX	benzene, toluene, ethylbenzene, xylene
bgs	below ground surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
CERCLIS	Comprehensive Environmental Response, Compensation, and Liability Information System
CLP	Contract Laboratory Program
COC	Chain of Custody
CRL	Central Regional Laboratory
D&D	Decontamination & Decommissioning
DERP	Defense Environmental Restoration Program
DNT	dinitrotoluene
DOD	U.S. Department of Defense
DQO	Data Quality Objective
EPA	Environmental Protection Agency
ERIIS	Environmental Risk Information & Imaging Service
ERNS	Emergency Response Notification System
FSP	Field Sampling Plan
FUDS	Formerly Used Defense Sites
gpm	gallons per minute
GSA	Government Services Administration
HNO ₃	Nitric Acid
IDW	Investigation-Derived Waste
INPR	Inventory Project Request
IT	IT Corporation
LeRC	Lewis Research Center
MDL	Method Detection Limit
MRL	Method Reporting Limit
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
msl	mean sea level
NACA	National Advisory Committee for Aeronautics
NASA	National Aeronautical and Space Administration
NIST	National Institute of Standard Technology
NPL	National Priorities List
OEPA	Ohio Environmental Protection Agency
PA	Preliminary Assessment
PAWL	Pentolite Area Waste Lagoons
PBOW	Plum Brook Ordnance Works
PBRF	Plum Brook Reactor Facility
PBS	Plum Brook Station
PE	Pentaerythritol

PETN	Pentaerythritol tetranitrate
PID	Photoionization Detector
ppb	parts per billion
PRGs	Preliminary Remediation Goals
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
SAIC	Science Applications International Corporation
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SAS	Special Analytical Services
SI	Site Investigation
SOP	Standard Operating Procedure
SVOC	Semi-Volatile Organic Compound
SW846	Test Methods for Evaluating Solid Waste 1986
TAL	Target Analyte List
TCL	Target Compound List
TOC	Total Organic Carbon
TNT	trinitrotoluene
USACE	United States Army Corps of Engineers
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

SECTION I

PROJECT DESCRIPTION

1.1 Introduction

The U.S. Army Corps of Engineers, Louisville District, has prepared this Quality Assurance Project Plan (QAPP) for a limited Site Investigation (SI) of the Pentolite Area Waste Lagoons (PAWL) site. The purpose of this limited Site Investigation (SI) is to evaluate the potential for contamination of the (PAWL) site due to past Department of Defense (DOD) activities. The potential for contamination at the former PAWL site was identified in an Inventory Project Request (INPR) (Huntington District U.S. Army Corps of Engineers (USACE), 1998). The References section of this document identifies the guidance documents and information used to evaluate the site history and environmental media and locations most likely to be affected. Field sampling and chemical laboratory analysis will be performed to evaluate the environmental media in the potential source area. Results of the laboratory analysis will be compared to risk-based, media specific screening criteria. USEPA Region 9 Preliminary Remediation Goals (PRGs) will be used as the screening criteria.

The PAWL is being addressed by the DOD under the Defense Environmental Restoration Program (DERP), Formerly Used Defense Sites (FUDS) program. An associated Field Sampling Plan (FSP) and Data Quality Objectives (DQOs) document, dated May 1999, are attached to this QAPP. A Site Specific Health and Safety Plan (SSHSP), date May 1999 is also associated with this document, but is not attached.

1.2 PBOW Installation History

Based on the Archives Search Report (USACE, 1993), the PBOW facility was established in 1941 and referred to as Plum Brook Ordnance Works (PBOW). The installation was established for the purpose of manufacturing trinitrotoluene (TNT), dinitrotoluene (DNT), pentolite, and nitric and sulfuric acids. Built by E.B. Badger and Sons Company, the facility was operated under contract by the Trojan Powder Company. Production of explosives ceased two weeks after V-J Day, having manufactured in excess of one billion pounds of explosives during the four-year operating period.

By September 1945, the entire Ordnance Inspection Department was abolished. Decontamination of TNT, acid, pentolite and DNT manufacturing lines was completed during the last quarter of 1945. On 17 December 1945, the physical custody of the plant was transferred from Trojan to the Ordnance Department. The U.S. Army Corps of Engineers assumed responsibility for maintenance and custodial duties until September 1946 when the property was transferred to the War Assets Administration (predecessor to the Government Services Administration (GSA)), after it was certified by the U.S. Army to be decontaminated. National Aeronautical and Space Administration (NASA) acquired the PBOW in 1963 and is presently using the site, now referred to as Plum Brook Station (PBS).

The PBS site currently lies in an area that is primarily rural and agricultural with a low population density. The NASA Glenn Research Center occupies a majority of the former ordnance works. The Department of the Army maintains a reserve center on the westernmost portion of the facility. The remainder of the former installation is in private ownership with the vast majority being cultivated. A tract on the northern boundary is owned by the Perkins Board of Education and is utilized as a bus maintenance facility

1.3 PAWL Site Description

1.3.1 General

The PAWL site is located within the northern portion of the former Plum Brook Ordnance Works (PBOW) facility in Erie County, Ohio (see Figures 1A and 1B). The former PBOW facility encompasses much of Perkins and Oxford townships approximately four miles south of Sandusky, Ohio.

The former PAWL includes approximately 1.3 acres within the currently existing Plum Brook Reactor Facility (PBRF) as shown on the Boring Location Plan (Figure 2). The PAWL are not currently detectable and the area is grass covered and relatively flat. This area is located just south of the PBRF Hot Laboratory and surrounded by an 8-foot tall chain-link fence. The distance from the PAWL to the nearest residence is approximately $\frac{3}{4}$ of a mile.

1.3.2 PAWL, Site Specific Operational History

Based on the PBS Preliminary Assessment (PA) (Science Applications International Corporation Report (SAIC), 1991), the first stage of the pentolite manufacturing process involved the nitration of pentaerythritol (PE) by adding nitric acid and water. Pentaerythritol tetranitrate (PETN) was an end product of this process, along with wastewater containing 5% nitric acid. Then, the PETN was treated with water, acetone, and ammonium bicarbonate to dissolve and neutralize the free acid present. Mixing equal parts of PETN and TNT produced pentolite. This step also involved the addition of acetone and water. The stoichiometric relationship of the pentolite manufacturing process is as follows:

- $PE + HNO_3 + H_2O \rightarrow PETN + \text{wastewater (nitric acid as 5\%)}$
- $PETN + \text{acetone} + \text{ammonia bicarbonate} + H_2O \rightarrow \text{preliminary filtered neutralized PETN} + \text{wastewater}$
- $(TNT + \text{acetone}) + (PETN + H_2O) \rightarrow \text{pentolite} + \text{wastewater}$

The wastewater from the pentolite manufacturing process potentially contained explosives, metals and acetone. Wastewater from each stage of the manufacturing process was conveyed to two settling basins or the PAWL via vitreous sewer pipes. A wastewater ditch (15 inch channel

pipe) encircled the entire perimeter of the lagoons. Wastewater from the first two pentolite lines, along with wastewater from the acetone recovery house, entered the lagoon at the west inlet to the wastewater ditch. Wastewater from the third pentolite line entered the lagoon at the east inlet to the wastewater ditch. Once inside the ditch, the flow of the wastewater was north to south. Three inverts, located at the north and south ends of the lagoons, fed the wastewater into two timber rising wells filled with limestone. These limestone beds were apparently filters and extended 35 ft. into the lagoons. It appears that the remainder of the basins further settled the wastewater until it eventually passed over the outlet weir and was pumped to a secondary treatment facility. Specific wastewater details, treatment processes, and sludge disposal methods are not known.

The PAWL were constructed of pre-cast concrete blocks (15 ft. by 9 ft. by 4 in. thick) with asphalt expansion joints, and each lagoon measured approximately 112 ft. by 137 ft. Four to six inches of No. 4 gravel had underlain the PAWL. Details of the Plans and Sections of the PAWL are shown on Figures 3A and 3B (attached).

Based on the Site Inspection Report (Morrison Knudsen, 1994), the Pentolite Area was decommissioned in 1945 when ordnance manufacturing ended, and all of the buildings in the area were supposed to be removed or burned in place according to the decontamination procedures. Decontamination of pentolite manufacturing lines was halted during the last quarter of 1945; and it was estimated that 65 % of the necessary decontamination of PBOW was completed by December 1945. The "Shut Down and Decontamination Procedures for PBOW, Sandusky, OH," (Dykema and Lee 1944) states that the stand by and storage procedure for the pentolite settling basin would have consisted of the following:

Drain and flush limestone bed and basin proper with a high-pressure hose.
Remove limestone while it is completely wet and flush basin thoroughly after removal.
Inspect basin thoroughly for evidence of accumulated explosives.

Based on the Records Reviews Report (Dames & Moore, 1997), there is no information regarding decontamination of surrounding soils in the Pentolite Area. Furthermore, the decontamination procedures do not mention the removal of the concrete slabs of the PAWL. An aerial photograph from 1956 shows that the lagoons were still intact. However, this aerial photo indicates that the vitreous sewer pipes leading to the lagoons were removed. From the decontamination procedures, it is assumed that these lines were flushed with water and then cleaned with acetone.

According to SAIC, the PAWL remained essentially intact until 5 July 1956 when approximately 500 acres (including the Pentolite Area and PAWL) was leased by the NASA from the Department of the Army to construct and operate the Plum Brook Reactor Facility. The reactor was planned to be a scientific investigation reactor where the effects of radiation on various materials could be measured. The entire Pentolite Area was filled and graded to facilitate construction of the PBRF. According to Dames and Moore, a memo by Everett and Campbell in 1958 states that the "Pentolite Area of approximately 117.3 acres was decontaminated, demolished, and cleared for use as the Lewis Laboratory Reactor Facility". A 1959 historic

topographic quadrangle reveals the PBRF with no evidence of the PAWL.

1.3.3 Facility/Size and Border

The PBOW is located near Sandusky within Erie County, Ohio (Figures 1A and 1B). The waste lagoons were located within the northern portion of PBOW, specifically, within the Pentolite Area, north of Pentolite Road and south of the present Plum Brook Reactor Facility (PBRF). The Layout of the PAWL is given on the Boring Location Plan (Figure 2).

The former PAWL includes approximately 1.3 acres within the currently existing PBRF. The PAWL are not currently detectable and the area is grass covered and relatively flat. This area is located just south of the PBRF Hot Laboratory and surrounded by an 8-foot tall chain-link fence.

The former PAWL area is bordered to the north by the PBRF, to the south by Pentolite Road and by access roads to the east and west.

1.3.4 Climate

The climate for Erie County is continental with cold and cloudy winters and warm and humid summers. The county's first freezing temperature is typically in October, and its last freezing temperature is typically in April. Average annual precipitation for Sandusky from 1961 to 1990 was 34.05 inches. Within that time period February had the lowest mean monthly rainfall average with 1.65 inches, whereas July had a high of 3.70 inches. The weather changes every few days as cold fronts move through the region. Wind is from the southwest 55 percent of the time (Morrison Knudsen 1994, Dames & Moore 1997).

1.3.5 Topography

According to historic plans obtained from the PBS Preliminary Assessment (PA) (Science Applications International Corporation Report (SAIC), 1991), the surface elevation of the waste lagoons was 626.15 feet msl. The perimeter of the waste lagoon angled at a slope of about 2:1 to a bottom elevation of 620.5 msl. The surface of the area that presently occupies the former PAWL is relatively flat and at about 630 feet above mean sea level (msl). Based on review of historic topographic quadrangles (USGS, 1959, 1969 and 1979), the surface elevation in the former PBOW has not changed significantly since 1959.

1.3.6 Geology

Based on the Site Wide Ground Study (IT Corporation, 1997-1998), three formations, all of the Devonian Age, underlie the former PBOW site. The Delaware Limestone is the lowermost formation. It is characterized as a hard, dense, finely crystalline limestone and dolomite.

Dissolution of this unit has been described which has produced solution channels along bedding planes and joints, and even producing caverns in some areas. The unit is typically buff colored and usually described as fossiliferous. In the vicinity of PBOW, benzene, toluene, ethyl benzene, and xylene (BTEX) and hydrogen sulfide are common in area quarries. Overlying the Delaware Limestone is the Olentangy Formation. Two members of the Olentangy Formation have been characterized at the PBOW site, the Plumbrook Shale and the overlying Prout Limestone. The Plum Brook Shale is interpreted to consist of approximately 35 feet of bluish-gray, soft, fossiliferous shale containing thin layers of dark, hard, fossiliferous limestone. The Prout Limestone has been interpreted to be a unit approximately 15 feet thick which outcrops occasionally in a 1,000 to 2,000 foot-wide, northeast striking band across the middle portion of the PBOW. It has been described as a dark-gray to blue, very hard, silicious, fossiliferous limestone or dolomitic mudstone. The uppermost formation at the PBOW site is the Ohio Shale. Only one member of the Ohio Shale is present in the PBOW area- the Huron Shale. This unit has been described as black, thinly bedded, with pyrite and abundant carbonaceous matter with some large pyrite/carbonate concretions up to 6 feet in diameter.

The bedrock overburden in Erie County is predominantly glacial till, glacial outwash or glacial lacustrine (lake) deposits. In the vicinity of PBOW, the soil has been interpreted to be lacustrine. In many areas, the overburden also consists of highly weathered bedrock. The thickness of the overburden ranges from approximately 5 feet or less for most of PBOW to greater than 25 feet. The overburden is thickest on the northern portion of the site.

A subsurface boring record (Dames and Moore, 1997), located about ¼ mile east of the PAWL site, indicated a subsurface profile consisting of an upper layer of brown fine sand extending from the ground surface to a depth of approximately 6 ft bgs. Based on the review of historical grading plans, this upper layer may be fill material associated with the construction of the PBRF. This fill may be over the PAWL site based on the historic grading plans reviewed. The presence of this fill is further indicated by review of the United States Department of Agriculture (USDA) Soil Conservation Service, Soil Survey of Erie county dated 1971. This upper layer is generally underlain by a layer consisting of silty clay and clayey silt soils extending to limestone bedrock. Shale fragments were encountered in the overburden soils from 14 feet to 24 feet bgs. Limestone bedrock was encountered at a depth of about 24 ft. bgs.

1.3.7 Hydrogeology

Based on the Site Wide Groundwater Study (IT Corporation 1997-1998), potable groundwater is encountered in the bedrock units underlying the PBOW site. Generally this groundwater flows northward toward Lake Erie. Based on published hydrogeologic information (Groundwater Resources of Erie County, 1986), the PBOW site includes 3 distinct hydrogeologic regions. Groundwater yields from these regions range from limited, to the northeast and south, to more than 500 gallons per minute (gpm), to the northwest.

It is anticipated that groundwater in the overburden soils beneath the PAWL site would be perched or trapped water. Perched water occurs in irregular, discontinuous granular zones within

the soil overburden. Perched water sources contain widely varying quantities of water depending on recent precipitation and other site-specific factors. Based on the soil types expected, overburden groundwater quantities should be minimal.

Based on the aforementioned hydrogeologic information, the PAWL site is in a hydrogeologic region that reports yields of 15 gpm, or less from wells drilled into the limestone. Hydrogen sulfide may be present in varying amounts.

1.4 Past Data Collection Activities and Current Status

No previous investigations have been performed at the waste lagoons. However, an Underground Storage Tank Corrective Actions Remedial Investigation, Feasibility Study, Phase I Report (Ebasco Environmental, 1991) was performed in the vicinity of the lagoons. Six monitoring wells were installed, and groundwater and soil samples were collected. Four VOCs were detected in the soil samples.

Based on reports and documents reviewed for the site, and a current assessment of all available information, the following target compounds and source area release mechanisms have been targeted for further investigation. Formal Data Quality Objectives (DQOs) for this SI were evaluated and documented in an associated attached document dated May 1999.

Based on historical operations at the PAWL, the COC that were potentially released to the environment include explosives, acetone, and metals. Therefore, samples will be analyzed for explosives, TAL metals, and VOCs. In addition, Total Organic Carbon (TOC) analysis, pH, and sieve and/or plasticity testing will be performed on selected samples in order to assess the soil characteristics at the site.

Based on historical construction drawings, the elevation at the bottom corners of the waste lagoons was about 620.5 ft. msl. The elevation of the top of the lagoons was about 626 ft msl. The ground surface in this area is presently at about 630 ft. msl.

The bottom of the waste lagoons consisted of a 4-inch thick concrete pad with 4 to 6 inches of gravel subbase. It is believed that the PAWL were removed during construction of the PBRF; however, there is a potential that the PAWL are still intact and fill material was simply placed over the lagoons during construction of the PBRF. Also, affected soils may have been redistributed to any elevation, including the ground surface, during these grading operations.

Based on the nature of the COC and expected grading operations associated with the decommissioning of the waste lagoons and construction of the PBRF, affected media, if present, would most likely be encountered within or just below the fill placed to achieve present grades.

1.5 Project Objectives

The purpose of this investigation is to gather sufficient information to evaluate the potential for environmental contamination at the site.

- Evaluate the existence of contamination in previously identified on-site, source area. Data quality must be sufficient to be able to compare with USEPA Region 9 health-based criteria (Preliminary Remediation Goals (PRGs)).
- Collect sufficient data on potentially contaminated media to support a recommendation for further study, interim action, or coordinate with the OEPA to proceed toward a No Further Action (NFA) closure of the site.

The investigation will integrate existing data with information that will be gathered through direct field investigations. The field investigation will include surface soil and subsurface soil sampling within the expected source area. Samples will be analyzed for volatile organics, explosives and Target Analyte List (TAL) metals. A limited number of samples will also be analyzed for Atterburg limits, percent moisture, grain size distribution, and total organic carbon (TOC) to determine soil physical parameters and their effect on contamination migration. Data from the investigation will be evaluated in conjunction with existing data to determine the next course of action. The rationale and scope of future activities will be discussed with and approved by the Ohio EPA prior to implementation.

If SI data suggests that site characterization information indicates that no further action is required at the site, activities will be generated with the OEPA to proceed toward a NFA Decision Document. If, on the other hand, the SI data shows a need for further investigation or other action, work plans for the next action will be prepared and submitted to OEPA.

1.5.1 Specific Objectives and Associated Tasks

For this project, it will be necessary to gather sufficient information to evaluate the existence of contamination due to potential releases from the PAWL. Some field monitoring will be utilized for purposes of screening for contaminants and for worker health safety. Field monitoring includes PID, TNT and Radionuclides.

In order to assess the presence or absence of hazardous constituents at the PAWL site, soil samples will be field screened during this SI for likely contaminants of concern, including volatile organics and TNT. In addition, chemical laboratory analysis for VOCs, explosives and TAL metals. A limited number of samples may also be analyzed for other soil characteristics. This information will be used to compare results to appropriate screening levels. The field monitoring requirements are detailed in the attached FSP.

1.5.2 Project Target Parameters and Intended Data Usages

The list of target parameters for this project includes VOCs, TAL metals and explosives. The data shall be compared to the USEPA Region 9 PRGs.

1.5.3 Quality Objectives and Criteria for Measurement Data

DQOs are qualitative and quantitative statements derived from outputs of each step of the DQO process that:

- Clarify the study objective;
- Define the most appropriate type of data to collect;
- Determine the most appropriate conditions from which to collect the data

The DQOs are then used to develop a scientific and resource-effective sampling design.

The DQO process allows decision-makers to define their data requirements and acceptable levels of decision during planning before any data are collected. DQOs are based on the seven step process described in EPA QA/G-4 (September 1994) document. The DQO process for this site is formally documented and attached to this QAPP.

1.6 Sample Network Design and Rationale

1.6.1 Sample Network by Task and Matrix

Sample matrices, analytical parameters and frequencies of sample collection can be found in the attached Field Sampling Plan.

1.6.2 Site Maps of Sampling Locations

The site location plan and proposed sampling locations are shown on Figures 1A, 1B and 2. It is possible, however, that depending on the nature of encountered field conditions some of these locations will be changed. The Site Field Manager, whose responsibilities are described in Section 2 of this QAPP, will be responsible for making such decisions.

1.6.3 Rationale of Selected Sampling Locations

The attached FSP and DQOs describe the rationale used to select sampling locations and depths. In general, sampling will be performed at locations most likely to contain the highest level of contaminants, that is, within and below the former lagoons.

1.7 Project Schedule

The earliest date for which sampling activities are planned is June 1999.

SECTION 2

PROJECT ORGANIZATION AND RESPONSIBILITY

This work is being performed by the United States Army Corps of Engineers (USACE) as part of the Defense Environmental Restoration Program (DERP)/ Formally Used Defense Sites (FUDS). The primary responsibility for the project lies with the Huntington District (CELRH) that acts as the administrator of the funds and performs the overall management functions. CELRH has tasked the Louisville District (CELRL) to execute the design, fieldwork and technical reporting. Several institutions and individuals will coordinate efforts to carry on the project. Their names and functions are listed below.

Industrial Hygienist:	Shelton Poole, CELRL-ED-EB
Project Engineer:	Chris Karem, CELRL-ED-EE
Safety QC:	Shirley Dunn, CELRL-SO
Risk Assessor:	David Brancato, CELRL-ED-EE
Hydrogeologist:	Martin Wahking, CELRL-ED-EB
QA Chemist:	Samir Mansy, CELRL-ED-EB
Independent Technical Review:	Doug Meadors, CELRL-ED-EE
Project Manager:	Rick Meadows, CELRH-DL-M

2.1 Project Organization

The lines of authority for this specific project are outlined below:

Shelton M. Poole, CHMM, RPIH Health and Safety Manager (HSM)

Mr. Poole has the responsibility for ensuring that the provisions of the Health and Safety Plan (HASP) are adequate and implemented in the field. Changing field conditions may require decisions to be made concerning the adequacy of the protection programs. Mr. Poole is well experienced and meets the additional training requirements specified by OSHA in 29 CFR 1910.120. The HSM is also responsible for conducting site inspections on a regular basis in order to ensure the effectiveness of the HASP.

Shirley Dunn Health and Safety Manager QC and Alternate

Ms. Dunn is well experienced and had the additional training requirements specified by OSHA in 29CFR1910.120. She will serve as the QC reviewer and alternative to Mr. Poole.

David Brancato, Ph.D., RPIH Risk Assessor

Dr. Brancato is well experienced in Risk Assessment methodologies.

Samir A. Mansy, Ph.D. Chemistry Quality Assurance Manager

Dr. Mansy served as the Chief of the Quality Assurance Section at Great Lakes and Ohio River Division Laboratory, Cincinnati, Ohio. He is currently the Data Quality Assurance Manager in Louisville District, Environmental Engineering Branch. He is experienced in data review, validation, and troubleshooting. Dr. Mansy provides an independent review of the analytical data based on SW846 and National Functional Guidelines.

2.2 Management Responsibilities

Site Investigation Project Manager

The Project Manager, Rick Meadows, Huntington District, has the overall responsibility for all phases of the PBOW projects.

Site Manager

The Project Engineer serves as the Site Manager. He is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The Site Manager's primary function is to ensure the successful achievement of technical, financial, and scheduling objectives. The Site Manager will:

- Define project objectives and develop a detailed work plan schedule;
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;
- Orient all field leaders and support staff concerning the project's special considerations;
- Monitor and direct the field team;
- Review the work performed on each task to ensure its quality, responsiveness and timeliness;
- Approve all reports (deliverables) before their submission to Ohio EPA;
- Ultimately be responsible for the preparation and quality of interim and final reports

The Project Engineer also acts as the contractor project manager and has overall responsibility for ensuring that the project meets the Corps of Engineers' objectives and quality standards. The Project Engineer/Scientist will provide assistance in writing and distributing the QAPP to all those parties connected with the project including the laboratory, Quanterra Environmental Services. The Project Engineer/Scientist is responsible for the technical quality control and

project oversight.

2.3 Quality Assurance (QA) responsibilities

Chemical QA Manager

The chemical QA Manager will remain independent of direct job involvement and day-to-day operations, and have direct access to corporate executive staff as necessary, to resolve any QA dispute. Dr. Mansy is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, U.S. Army Corps of Engineers, and Ohio EPA requirements. Specific functions and duties include:

- 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 Project Engineer.
- Data validation including tentatively identified compounds;
- Review and approval of field and laboratory procedures.
- Performance and system Audits of the Laboratory.

All samples will be analyzed by Quanterra Environmental Services, North Canton, Ohio, with the exception of Atterburg Limits and grain size analysis, which will be subcontracted to an approved laboratory. Data validation will be done by Roy F. Weston, Inc., Miamisburg, Ohio. Validation will be conducted randomly on 10% of the sample results.

2.4 Field Responsibilities

USACE Field Leader

The Project Engineer will act as the field team leader. He is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. The USACE field team leader is an environmental professional and will report directly to the Project Manager. Specific field-team leader responsibilities include:

- Provision of day-to-day coordination with the Project Manager on technical issues in specific areas of expertise;
- Developing and implementing of field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements;
- Coordinating and managing of field staff including sampling, drilling, and supervising

field laboratory staff;

- Acting as field sample custodian;
- Implementing of QC for technical data provided by the field staff including field measurement data;
- Adhering to work schedules provided by the Project Manager;
- Authoring, writing, and approving of text and graphics required for field team efforts;
- 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 Project Manager, implementing and documenting corrective action procedures, and provision of communication between team and upper management; and
- Participating in preparation of the final report.

USACE Field Technical Staff

The technical staff for this project will be drawn from USACE pool of Louisville District, Environmental Engineering Branch resources. The technical team staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

2.5 Laboratory Responsibilities

Quanterra Laboratory Project Manager

The Quanterra Project Manager, Debora Hula, will report directly to the USACE Quality Assurance Manager, Dr. Samir Mansy, and will be responsible for the following;

- Ensuring all resources of the laboratory are available on an as-required basis;
- Providing overview of final analytical reports; and
- Approving final analytical reports prior to submission to Louisville District.

Quanterra Operations Manager

The Quanterra Operation Manager will report to the Quanterra Project Manager and will be

responsible for:

- Coordinating laboratory analyses;
- Supervising in-house chain-of-custody;
- Scheduling sample analyses;
- Overseeing data review; and
- Overseeing preparation of analytical reports.

Quanterra Quality Assurance Officer

Ms. Opal Davis-Johnson is the Quanterra QA Officer, and has the overall responsibility for data after it leaves the laboratory. The Quanterra QA Officer will be independent of the laboratory but will communicate data issues through the Quanterra Project Manager. In addition, the Quanterra QA Officer will:

- Overview laboratory quality assurance;
- Overview QA/QC documentation;
- Conduct random audits of detailed data;
- Determine whether to implement laboratory corrective actions, if required;
- Define appropriate laboratory QA procedures;
- Prepare laboratory Standard Operating Procedures; and
- Sign the title page of the QAPP.

Quanterra Sample Custodian

The Quanterra sample custodian, Lois Ezzo, will report to the Quanterra Operations Manager, Ms. Debora Hula, and to the Laboratory Supervisor. Responsibilities of the sample custodian will include:

- 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 laboratory manager and laboratory supervisor of sample receipt and inspection;
- Assigning a unique identification number and customer number, and entering each into the sample receiving log;
- With the help of the laboratory manager, initiating transfer of the samples to appropriate lab sections; and
- Controlling and monitoring access/storage of samples and extracts.

Final responsibility for project quality rests with USACE Project Manager. Independent quality assurance will be provided by the Quanterra Project Manager and QA Officer prior to release of all data to the USACE Project Engineer/Scientist .

Quanterra Technical Staff

The Quanterra technical staff will be responsible for sample analysis and identification of corrective actions. The staff will report directly to the Quanterra Operations Manager. The technical staff have signed Ethics Agreements which state: they will abide by the high standards of integrity; they shall report actual data; and they will report to the officials of any accidental or intentional non-authentic data. Copies of the agreements are included in Appendix A.

SECTION 3

QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results that are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventative maintenance of field equipment, and corrective action are described in other sections of this QAPP.

3.1 Precision

3.1.1 Definition

Precision is a measure of the degree to which two or more measurements are in agreement.

3.1.2 Field Precision Objectives

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 analytical samples.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent difference (RPD) and relative standard deviations (RSD) for three or more samples. The equations to be used for precision in this project can be found in section 12 of this QAPP. Precision control limits are included in the provided SOPs.

3.2 Accuracy

3.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value.

3.2.2 Field Accuracy Objectives

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.

3.2.3 Laboratory Accuracy Objectives

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 12 of this QAPP. Accuracy control limits are included in the provided SOPs.

3.3 Completeness

3.3.1 Definition

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

3.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project.

3.3.3 Laboratory Completeness Objectives

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 12 of this QAPP. Laboratory completeness for this project will be greater than 95 percent.

3.4 Representativeness

3.4.1 Definition

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.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP is followed and that proper sampling techniques are used.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample-holding times and analyzing and assessing field duplicated samples. The sampling network is designed to provide data representative of facility conditions. During development of this network, consideration is given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the FUDS program. The rationale of the sampling network is discussed in detail in the FSP.

3.5 Comparability

3.5.1 Definition

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP is followed and that proper sampling techniques are used.

3.5.3 Measures to Ensure Comparability of Laboratory Data

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.

3.6 Level of Quality Control Effort

Field blank, trip blank, method blank, duplicate, standard reference materials (SRM) and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Field and trip blanks consisting of distilled water will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the facility, which may cause samples contamination. Trip blanks are used to assess the potential for contamination of samples due to contamination migration during sample shipping and storage. Trip blanks generally pertain to volatile organic samples only. Trip blanks prepared prior to the sampling event in the actual sample containers and are kept with the investigative samples throughout the sampling event. They are then packaged for shipment with other samples and sent for analysis. There should be one trip blank included in each sample-shipping container. At no time after their preparation are the sample containers opened before they reach the laboratory.

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. Matrix spikes provide information about the effect of sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One matrix spike/matrix spike duplicate will be collected for every 20 or fewer investigative samples. MS/MSD samples are designated/collected for organic analyses only.

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOCs or extractable organics. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, soil).

The general level of the QC effort will be one field duplicate and one field blank for every 10 or

fewer investigative samples. One volatile organic analysis (VOA) trip blank consisting of distilled deionized ultra pure water will be included along with each shipment of aqueous VOA samples.

The number of duplicate and field blank samples to be collected is listed in the Field Sampling Plan. Sampling procedures are also specified in the Field Sampling Plan.

SECTION 4

SAMPLING PROCEDURES

The sampling procedures to be used in this site investigation will be consistent for the purpose of this project. The attached Field Sampling Plan outlines all the sampling procedure information. Please refer to the FSP for the following information:

1. Summary of Sampling Activity
2. Sampling Network Design and Rationale
3. Sampling Custody Procedure
 - a. Sample Identification Procedure
 - b. Initiation of Field Custody Procedure
 - c. Field Activity Documentation / Logbook
 - d. Sample Shipment and Transfer of Custody
4. Sample Containers, Sample Preservation, and Maximum holding time.
5. Sample handling, Packaging and shipment
6. Decontamination Procedures
 - a. Personnel and Equipment
 - b. Sample Bottles
 - c. Sampling Devices
7. Sampling Equipment and Procedures
 - a. Soil Sampling Procedures
 - * Sampling Devices
 - * Sampling Procedures
8. QC Sample Procedures
9. Field Blank Sample Collection

Field Duplicate Sample Collection

11. Matrix Spike / Matrix Spike Duplicate Sample Collection
12. Trip Blank Sample Preparation

13. Field Measurement / Screening
14. Preventative Maintenance Procedure / Schedule
15. Storage and Disposal of Investigative Derived Waste (IDW)

SECTION 5

CUSTODY PROCEDURES

Custody is a vital factor necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if:

- * the item is in actual possession of a person; or
- * the item is in the view of the person after being in actual possession of the person; or
- * the item was in actual physical possession but is locked up to prevent tampering; or
- * the item is in a designated and identified secure area.

5.1 Field Custody Procedures

Field logbooks will provide the means of recording data collecting activities performed. 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. Each logbook will be identified by the project-specific document number.

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

Measurements made and samples collected will be recorded. 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 which is 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 which includes compass and distance measurements, shall be recorded. The number of photographs taken of the station will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

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 tags with sample numbers, sampling locations, date / time of collection, and type of analysis.
- c) Sample tags are to be completed for each sample using waterproof ink unless inhibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.
- d) Samples are accompanied by a properly completed chain of custody form. 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 samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.
- e) 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 locked and 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 strapped shut with strapping tape in at least two locations.
- f) All shipments will be accompanied by the Chain of Custody Record identifying the contents. The cooler number will be entered on the Chain of Custody record. The original record will accompany the shipment, and a copy will be retained by the sampler for returning to the sampling office.
- g) If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.
- h) Samples will be transported to the laboratory the same day the samples are collected in the field by overnight carrier.

5.2 Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and log-in; sample storing and numbering; tracking during sample preparation and analysis; and storage of data are described in the Quanterra procedures in Appendix B. Examples of laboratory chain of custody traffic reports along with instructions for completion are included in the Appendix.

The chain of custody procedures for samples shipped to the CRL are described in Quanterra Standard Operating Procedures (SOP).

5.3 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 QAPP. The Project Engineer is the custodian of the evidence file and maintains the contents of evidence files for the site, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secured, limited access area and under custody of the Site Manager.

The final evidence file will include at a minimum:

- field logbooks
- field data and data deliverables
- photographs
- drawings
- soil boring logs
- laboratory data deliverables
- data validation reports
- data assessment reports
- all custody documentation (tags, forms, Air bills, etc.)

SECTION 6

CALIBRATION PROCEDURES AND FREQUENCY

This section describes the calibration procedures and the frequency at which these procedures will be performed for both field and laboratory instruments.

6.1 Field Instrument Calibration

The field instruments will be calibrated as described by the manufacturer's procedures. Field instruments include a photoionization detector (PID) and a direct read radionuclide detector. As a rule, the calibration of the instruments will be checked daily and recalibrated when required.

6.2 Laboratory Instrument Calibration

Calibration procedures for a scientific laboratory instrument will consist of an initial calibration (2, 3, 5, or 6 points, depending on the method), initial calibration verification and continuing calibration verification. For a description of the calibration procedures for a specific laboratory instrument, refer to the applicable SOPs in Appendix B of this QAPP. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require recalibration. In all cases, the initial calibration will be verified using an independently prepared calibration verification solution (CRI-brand as second source).

The laboratory maintains a sample logbook for each instrument which will contain the following information: instrument identification, date of calibration, analyst, calibration solutions run and the samples associated with these calibrations.

Organic Analyses

Prior to calibration, the instrument(s) used for Gas Chromatographic / Mass Spectrometer (GC / MS) analyses are tuned by analysis of p-bromofluorobenzene (BFB) for volatile analyses and decafluorotriphenyl phosphine (DFTPP) for semivolatile analyses. Once the tuning criteria for these reference compounds are met, the instrument should be initially calibrated by using a five point calibration curve. The instrument tune will be verified each 12 hours of operation.

Continuing calibration is verified as specified in the method, or at least each working day, using criteria specified by the method. The calibration standards will be USEPA- or NBS-traceable and are spiked with internal standards and surrogate compounds. Whereas, calibration and continuing calibration verification at midpoint and at MRL (Method Reporting Limit) levels will be performed at approved intervals as specified by the manufacturer or the analytical method (whichever is more frequent). Calibration standards used as reference standards will be traceable to the source.

Metals Analysis

The Atomic Absorption Spectrophotometer (AAS) and Inductively Coupled Plasma Emission Spectrophotometer (ICP) instruments are calibrated by use of a blank and a one-point standard prepared by dilution of certified stock solutions. An analysis blank is prepared with one calibration standard at the MRL for the metal. The other standards bracket the concentration range of the samples. Calibration standards will contain acids at the same concentration as the digestates.

A continuing calibration standard, prepared from a different stock solution than that used for preparation of the calibration standards, is prepared and analyzed after each ten samples or each two hours of continuous operation. The value of the continuing calibration standard concentration must agree with ± 10 percent of the initial value or the appropriate corrective action is taken which may include recalibrating the instrument and reanalyzing the previous ten samples.

For the ICP, linearity near the reporting limit will be verified with a standard prepared at a concentration at the reporting limit (MRL >3MDL). This standard must be run at the beginning and end of each sample analysis run or a minimum of twice per 8-hour period.

SECTION 7

ANALYTICAL PROCEDURES

Samples will be analyzed by Quanterra Incorporated, North Canton, Ohio.

7.1 Field Analytical Procedures

The standardization and QA information for field measurements are described in Section 3 of this QAPP. A copy of the Field Sampling Plan is attached to this QAPP.

7.2 Laboratory Analytical Procedures

The laboratory named above will implement the project required Standard Operating Procedures (SOPs). These laboratory SOPs for sample preparation, cleanup and analysis are based on SW-846 Revision (Latest Version). These SOPs provide sufficient details and are applicable to this investigation.

The site samples for volatile organic compounds analysis (VOC) shall be screened in the laboratory, as described in the VOC SOP and shall be analyzed, either as low or medium level concentration samples, or as a series of dilutions in order to cover the expected concentration range of the site-specific compounds of interest.

The tables in Appendix E summarize the analyte groups of interest, appropriate laboratory SOP numbers and EPA reference method for the organic and inorganic analytes, respectively, to be evaluated in this investigation. The Quanterra SOPs to be used in this investigation are contained in Appendix B of this document.

7.3 List of Project Target Compounds and Laboratory Detection Limits

A complete listing of project target compounds, project quantitation limits, Method Reporting Limit (MRL), and current laboratory determined detection limits for each analyte group can be found in Appendix E of this QAPP. Method detection limits shown above have been experimentally determined using the procedure found in 40CFR, Part 136, Appendix B, or equivalent statistical approach. The latest MDLs at the time of sample analysis will be used.

SECTION 8

INTERNAL QUALITY CONTROL CHECKS

8.1 Field Quality Control Checks

QC procedures for field screening instruments are described in Section 6.0 of this QAPP. Assessment of field sampling precision and bias will be made by collecting field duplicates and field blanks for laboratory analysis. Collection of the samples will be in accordance with the procedures outlined in section 5.0 of the attached FSP.

8.2 Laboratory Quality Control Checks

The laboratory identified in Section 7 of this QAPP has a QC program it uses to ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as SOPs and each SOP includes a QC section, which addresses the minimum QC requirements for the procedure. The internal quality control checks might differ slightly for each individual procedure but in general the QC requirements include the following:

- Field /Trip blanks
- Method blanks
- Reagent/preparation blanks (applicable to inorganic analysis)
- Instrument blanks
- Matrix spikes/matrix spike duplicates
- Surrogate spikes
- Analytical spikes (Graphite furnace)
- Field duplicates
- Laboratory duplicates
- Laboratory control standards
- Internal standard areas for GC/MS analysis; control limits
- Mass tuning for GC/MS analysis

For a description of the specific QC requirements of this site investigation and the frequency of audit, refer to the laboratory SOPs. The QC criteria are also included in the SOPs.

All data obtained will be properly recorded. The data package will include a full deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. Any samples analyzed in nonconformance with 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.

SECTION 9

DATA REDUCTION, VALIDATION, AND REPORTING

All data generated through field activities or by the laboratory operation shall be reduced and validated prior to reporting. No data shall be disseminated by the laboratory until it has been subjected to the procedures summarized below.

9.1 Data Reduction

9.1.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. A PID, portable direct read radionuclide detector and TNT screening kit will generate field measurements. Such data will be written into field log books immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry.

9.1.2 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 (paper or electronic form). 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 tag number. Other details will also be recorded in the lab notebook, such as the analytical method used (SOP#), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook shall be signed and dated by the analyst. Copies of the strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the lab QA Manager takes place at the opening and closing of laboratory logs, at a minimum. (Records of notebook entry inspections are maintained by the QA Manager.)

All calculations are checked by the Organic, and Inorganic including Metal Section Supervisor at the conclusion of each operating day. Errors are noted, corrections are made, but the original notations are crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis.

Quality control data (e.g. laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. In Level 1 review, the analyst reviews all of the data and QC. This is followed by Level 2 review, in which a senior analyst reviews 100% of QC and 10% of the raw data. Data considered to be acceptable will be entered into the laboratory computer system. The computer system compares QC data to internally generated limits (LCS < MS/MSD, and surrogate) and method criteria. The data are logged into the project database. Unacceptable data shall 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 Lab Project Manager approves these data, they are considered ready for third party data validation.

9.2 Data Validation

Data validation procedures shall be performed for both field and laboratory operations as described below:

9.2.1 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. This task will be the responsibility of the Field Manager.

9.2.2 Procedures to Validate Laboratory Data

USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic and Inorganic Data Review, February 1994, procedures will be modified to include SW846 criteria summarized in Appendix C, Laboratory Analysis Criteria. The modified NFG will be followed to validate laboratory data in conjunction with the Data Validation Checklist found at Appendix D.

Roy F. Weston assessment will be accomplished by the joint efforts of the Data Reviewer and Project Manager. The data assessment by the Project Manager will be based on the criteria that the sample was properly collected and handled according to the field Sampling Plan and Section 5 of this QAPP.

The Roy F. Weston Data Reviewer will conduct a systematic review of the data for compliance with the established QC criteria based on the spike, duplicate and blank results provided by the laboratory. All technical holding times shall be reviewed, the GC/MS instrument performance check sample results shall be evaluated, results of initial and continuing calibration will be reviewed and evaluated by trained reviewers independent of the laboratory. Also, results of all blanks, surrogate spikes, matrix spikes/matrix spike duplicates, laboratory control samples, internal standards, target compound identification and quantitation, tentatively identified compounds, and system performance checks shall be performed for volatile organic compounds by the validator. Additionally, documents of method detection limits study will be provided to the validator. The results shall also be validated. One hundred percent of the data will be evaluated and/or validated.

The Data Review will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analysis may be made by the Project Engineer/Project Scientist based on the extent of the deficiencies and their importance in the overall context of the project.

All data generated for the 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 in

accordance with the Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (February 1994) and Inorganic Analyses (February 1994), as well as additional comments of the Data Reviewer. The laboratory-provided data flags will include such items as: concentration below required detection limit; estimated concentration due to poor spike recovery; and concentration of chemical also found in laboratory blank.

The Data Reviewer comments will indicate that the data are:
useable as a quantitative concentration;
useable with caution as an estimated concentration; or
3) unusable due to out-of-control QC results.

All CLP forms summarizing this information will be checked as well. The overall completeness of the data package will also be evaluated by the Data Validator. Completeness checks will be administered on all data to determine whether deliverables specified in the SI Work plan and 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. The reviewer will determine whether all required items are present and request copies of missing deliverables.

9.3 Data Reporting

Data reporting procedures shall be carried out for field and laboratory operations as indicated below:

9.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration checks.

9.3.2 Laboratory Data Reporting

Laboratory data is not considered official, reportable data until after the validation activity has been concluded via the laboratory QA Officer/Manager. The Laboratory Project Manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the record of chain-of-custody, the report format shall consist of the following:

1. 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 utilized and also 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 which may have created analytical difficulties

Discussion of any laboratory quality control checks which failed to meet project criteria

Tables summarizing QC checks for MRLs (true values, found values, and % recoveries) in CLP form

- Signature of the laboratory QA Manager

2. Chemistry Data Package

- Case narrative for each package/analytical group
- 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
- Results of (dated) initial and continuing calibration checks, and GC/MS tuning results
- Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results
- Labeled (and dated) chromatograms/spectra of sample results and laboratory quality control checks
- Results of tentatively identified compounds

The Data package will be a "CLP-like" format consisting of all the information presented in a CLP data package.

SECTION 10

PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of laboratory activities will be conducted to verify that analyses are performed in accordance with the procedures established in the FSP and QAPP. The audits of laboratory activities include two independent parts: internal and external audits.

10.1 Laboratory Performance and Systems Audits

The Quanterra Analytical Services laboratories are audited on a regular basis by U.S. Army Corps of Engineers. The U.S. Army Corps of Engineers Center of Expertise in Omaha, Nebraska conducts the system audits of the laboratories on an annual basis, and conducts performance audits.

The system audits, include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain of custody procedure, sample preparation and analysis, instrument operating records, etc. The performance audits will consist of sending performance evaluation (PE) samples to laboratories for on-going assessment of laboratory precision and accuracy. The analytical results of the analysis of PE samples are evaluated by U.S. Army Corps of Engineers Center of Expertise to ensure the laboratories maintain good performances.

10.1.1 Internal laboratory Audits

10.1.1.1 Internal Lab Audit Responsibilities

The internal laboratory audit will be conducted by the Quanterra QA Officer.

10.1.1.2 Internal Lab Audit Frequency

The internal lab system audits will be done on an annual basis while the internal lab performance audits will be conducted on a quarterly basis.

10.1.1.3 Internal Lab Audit Process

The internal lab 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. The Quanterra QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance. The laboratory audit checklist has been submitted.

10.1.2 External Laboratory Audits

10.1.2.1 External Lab Audit Responsibilities

An external audit may be conducted by the Corps of Engineers.

10.1.2.2 External Lab Audit Frequency

An external lab audit may be conducted at least once prior to the initiation of the sampling and/or during analysis activities. These audits may or may not be announced and are at the discretion of the U.S. Army Corps of Engineers, Louisville District.

10.1.2.3 Overview of the External Lab Audit Process

External lab audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

SECTION 11

PREVENTATIVE MAINTENANCE

11.1 Field Instrument Preventive Maintenance

The field equipment for this project includes a PID, direct read radionuclide detector and a TNT test kit. Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked daily for calibration and calibrated if necessary. Calibration checks will be documented in the field logbooks. Backup instruments and equipment will be available on-site or within 1 day shipment to avoid delays in the field schedule.

11.2 Laboratory Instrument Preventive Maintenance

As part of their QA/QC Program, a routine preventive maintenance program is conducted by a service contractor on a limited basis to minimize the occurrence of instrument failure and other system malfunctions. Quanterra Laboratories personnel perform routine scheduled maintenance, and repair or coordinate with the vendor for the repair of all instruments. All laboratory instruments are maintained in accordance with manufacturer's 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 manufacturer's maintenance is provided under a repair and maintenance contract with factory representatives.

SECTION 12

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

12.1 Accuracy Assessment

In order 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 spike sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Control charts will be plotted periodically for each commonly analyzed compound and kept on method-specific, matrix-specific, and analyte-specific bases. The percent recovery for a spiked sample is calculated according to the following formula:

$$\%R = \frac{\text{Amount in spiked sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \times 100$$

12.2 Precision Assessment

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 of the aliquots 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 determine 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 and plotted. The RPD is calculated according to the following formula:

$$RPD = \frac{\left| \frac{(\text{Amount in Spike\#1} - \text{Amount in Spike\#2})}{(\text{Amount in Spike\#1} + \text{Amount in Spike\#2})} \right|}{2} \times 100$$

Control Charts for recoveries (%), and RPDs will be submitted with the data packages to the U.S. Army Corps of Engineers, Louisville District.

12.3 Completeness Assessment

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:

$$\text{Completeness} = \frac{(\text{number of valid measurements})}{(\text{number of measurements planned})} \times 100$$

SECTION 13

CORRECTIVE ACTION

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of quality control performance which 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 implemented after approval by the Project Engineer, or his designee. If immediate corrective action is required, approvals secured by telephone from the Engineer should be documented in an additional memorandum.

For noncompliance problems during laboratory analysis, 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 contractor's laboratory Project Manager, who in turn will notify the U.S. Army Corps of Engineer Quality Assurance Manger. 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 Field Sampling Plan will be identified and corrected in accordance with the QAPP. The U.S. Army Corps of Engineers Quality Assurance Manager, or his designee, will issue a nonconformance report for each nonconformance condition.

Corrective actions will be implemented 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 by stop-work order by the Quality Assurance Manager.

13.1 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 QAPP, etc.), sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Project Engineer. The Project Engineer will be responsible for assessing the suspected problems in consultation with the project QA Manager on making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, a nonconformance report will be initiated by the Project Engineer.

The Project Engineer will be responsible for ensuring that corrective action for nonconformances are initiated by:

- evaluating all reported nonconformances;

- controlling additional work on nonconforming items;
- determining disposition or action to be taken;
- maintaining a log of nonconformances;
- reviewing nonconformance reports and corrective actions taken;
- ensuring nonconformance reports are included in the final site documentation in project files.

If appropriate, the Project Engineer will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed. Corrective action for field measurements may include:

- Repeat the measurement to check the error;
- Check for all proper adjustments for ambient conditions such as temperature;
- Check the batteries;
- Re-Calibration:
- Check the calibration;
- Replace the instrument or measurement devices;
- Stop work (if necessary).

The Field Team Leader or his designee is responsible for all site activities. In this role, the Field Team Leader at times is required to adjust the site programs to accommodate site specific needs. When it becomes necessary to modify a program, the responsible person notifies the Field Team Leader of the anticipated change and implements the necessary changes after obtaining the approval of the Field Team Leader. The Field Team Leader must approve the change in writing or verbally prior to field implementation, if feasible. If unacceptable, the action taken during the period of deviation will be evaluated in order to determine the significance of any departure from established program practices and action taken.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The Quality Assurance Officer will identify deficiencies and recommended corrective action to the Project Manager. Implementation of corrective actions will be performed by the field team. Corrective action will be documented.

Corrective actions will be implemented 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 by the U.S. Army Corps of Engineers QA Manager.

13.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, 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 prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the laboratory Quality Control Coordinator to approve the implementation of corrective action. The submitted standard operating procedures (SOPs) specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic reinjection/reanalysis when certain quality control criteria are not met, etc. A summary of method-specific corrective actions is found in this QAPP.

Corrective action is implemented at several different levels. The laboratories are required to have a written SOP specifying corrective action to be taken when an analytical error is discovered or the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification by the analyst about the errors and corrective procedures. The Corps of Engineers also may request corrective action for any contractual nonconformance identified by audits or data validation. The COE may request corrective action by the laboratories for any nonconformances identified in the data validation process or, for minor problems, the lab may be contacted directly. Corrective actions may include:

- Re-analyzing the samples, if a holding time criterion permits;
- Resampling and analyzing, and/or;
- Evaluation and amending sampling procedures and/or
- Evaluation and amending analytical procedures; and/or
- Accepting data and acknowledging the level of uncertainty.

If resampling is deemed necessary due to laboratory problems, the Project Engineer must identify the necessary approach for the additional sampling effort.

Corrective actions are required whenever an out-of-control event or potential is noted. The investigative action taken is somewhat 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;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or 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.

These corrective actions are performed prior to release of the data from the laboratory. The corrective actions will be documented in both the 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 data validator. If corrective action does not rectify the situation, the laboratory will contact the Corps of Engineers QA Manager.

13.3 Corrective Action During Data Validation and Data Assessment

The U.S. Army Corps of Engineers may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (e.g. the holding time for samples is not exceeded, etc.). When the U.S. Army Corps of Engineers data assessor identifies a corrective action situation, the Project Manager will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions of this type will be documented by the QA Manager.

SECTION 14

QUALITY ASSURANCE REPORTS TO MANAGEMENT

The laboratory Project Manager will be responsible for deliverables associated with the tasks identified in the Work Plan. The Quality Assurance Officer will be responsible for reporting on the accuracy, precision, and completeness of the data as well as the results of the performance and system audits, and any corrective action needed or taken during the project.

14.1 Contents of Project QA Reports

The QA reports will contain on a routine basis all results of field and laboratory audits, all information generated during the past month reflecting on the achievement of specific data quality objectives, and a summary of corrective action that was implemented, and its immediate results on the project. The status of the project with respect to the Project Schedule included in the QAPP will be determined. Whenever necessary, updates on training provided, changes in key personnel, anticipated problems in the field or lab for the upcoming month that could bear on data quality along with proposed solutions, will be reported. Detailed references to QAPP modifications will also be highlighted. All QA report will be prepared in written, final format by the laboratory Project Manager or his designee. In the event of an emergency, or in case it is essential to implement corrective action immediately, QA reports can be made by telephone to the appropriate individuals, as identified in the Project Organization or Corrective Action sections of this QAPP. However, these events, and their resolution will be addressed thoroughly in the final QA report.

14.2 Frequency of QA Reports

Based on the short duration of this project, only one QA Report is anticipated and will be prepared at the end of the project. The report will continue without interruption, until the project has been completed. The frequency of any emergency reports that must be delivered verbally cannot be estimated at the present time.

14.3 Individuals Receiving/Reviewing QA Reports

The Project Engineer will orchestrate distribution of all QA reports.

References

Dames & Moore, Inc., April 1995, *Records Review Report, Plum Brook Ordnance Works, Sandusky, Ohio.*

Dames & Moore, Inc., April 1997, *Site wide Groundwater Investigation, Final Report Plum Brook Ordnance Works Plum Brook Station/NASA, Sandusky, Ohio*

Dykema, Henry J., and Lee, Harold S., March 1944, *Shut Down and Decontamination Procedures for Plum Brook Ordnance Works, Sandusky, Ohio, Ordnance Department.*

Ebasco Environmental, 1991, *Underground Storage Tank Corrective Actions Remedial Investigation, Feasibility Study, Phase I Report, November.*

E.B. Badger & Sons Co., November 9, 1942, "Job 2230 Pentolite Waste Water Disposal Collection System", Drawing No. 2239-P-109.

Environmental Protection Agency (EPA), September 1994, *Guidance for the Data Quality Objectives Process.*

Environmental Protection Agency (EPA), September 1992, *Guidance for Performing Site Inspections Under CERCLA.*

Environmental Risk Information & Imaging Services (ERIIS), May 1999, *Database Search of "Listed Facilities".*

International Technology (IT) Corporation, December 1997, *Site Investigation of the Reservoir No. 2 Burning Ground, Additional Burning Ground, Wastewater Disposal Plant No. 2, Powerhouse No. 2. Ash Pit, Plum Brook Ordnance Works, Sandusky, Ohio*

IT Corporation, 1999, *Site-Wide Groundwater Investigation, Former Plum Brook Ordnance Works, Sandusky, Ohio.*

International Consultations Incorporated (ICI), 1995, *Site Management Plan, Plum Brook Ordnance Works, Sandusky, Ohio, prepared for the U.S. Army Corps of Engineers, Huntington District, September.*

Morrison Knudsen Corporation (MK), 1994, *Site Inspection Report, Plum Brook Station, Sandusky, Ohio, Volume I*, (prepared for NASA), January.

Plum Brook Ordnance Works (PBOW), 1993, *Archives Search Report*, USACE
St. Louis District.

Science Applications International Corporation (SAIC), 1991, *Plum Brook Station Preliminary Assessment, Volume I, Area IV*.

U.S. Army Corps of Engineers, September 1994, *EM 200-1-3, Requirements for the Preparation of Sampling & Analysis Plans*.

U.S. Department of Agriculture, February 1971, *Soil Survey, Erie County, Ohio*, Soil Conservation Service.

War Department, 1945, *Decontamination Procedures*, War Department Supply Bulletin SB 5-52, July.

U.S. Department of the Interior, Fish and Wildlife Services, 1987, *National Wetlands Inventory Map*.

Figures

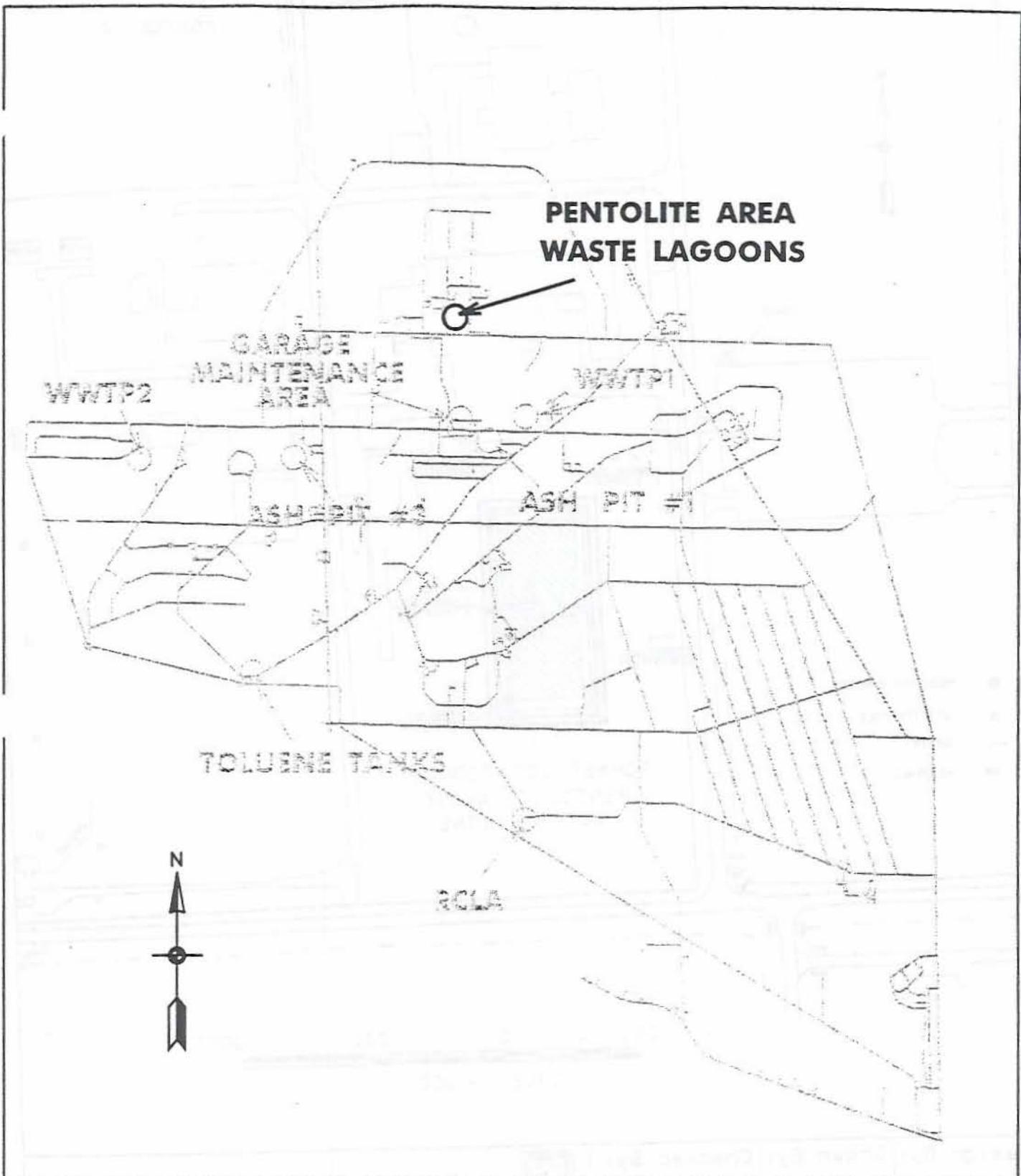
Figure 1A: PBOW Site Location Plan

Figure 1B: PAWL Site Location Plan

Figure 2: Boring Location Plan

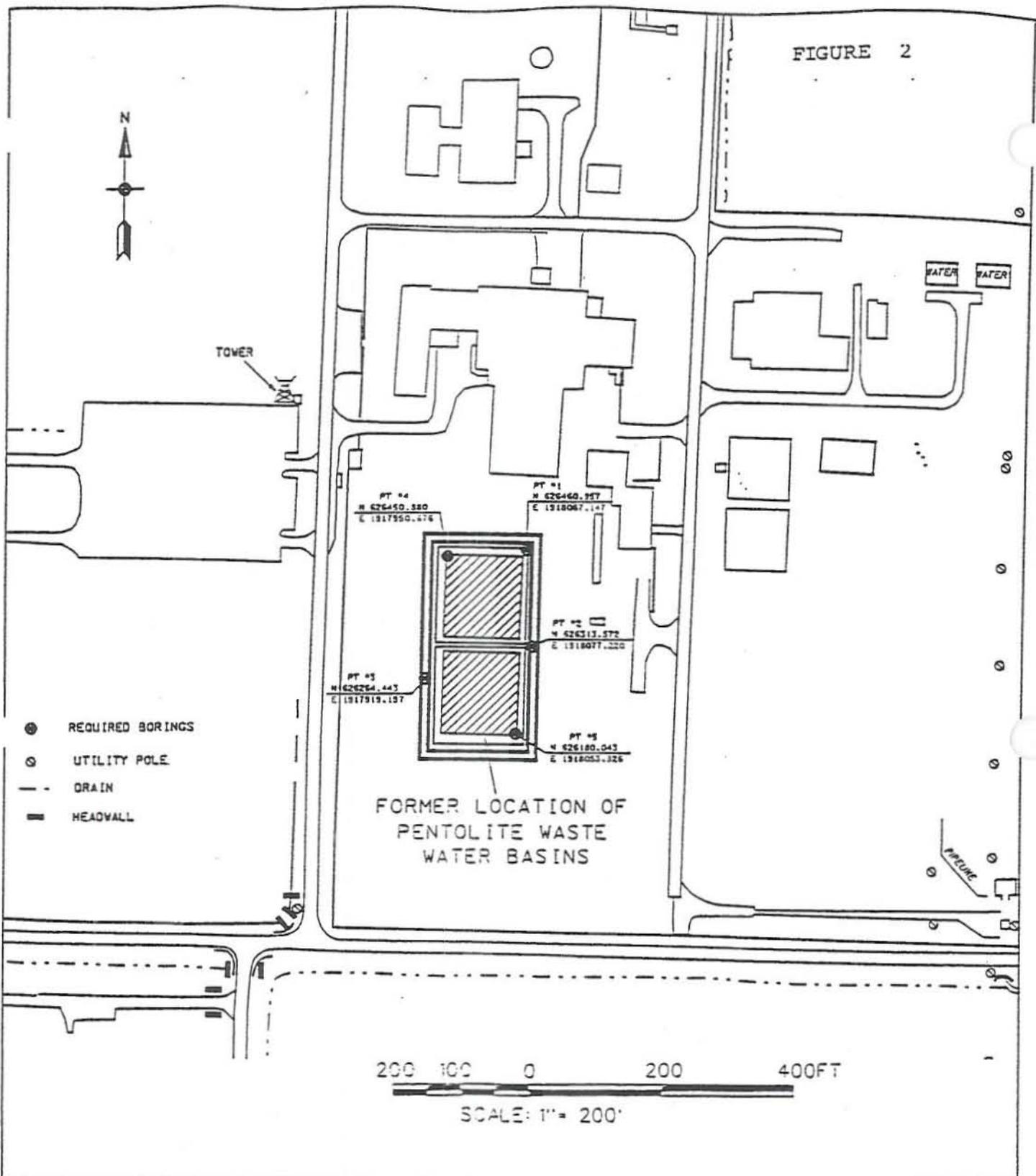
Figure 3A: Layout of Pentolite Waste Water Basins

Figure 3B: Construction Details of Pentolite Waste
Water Basins



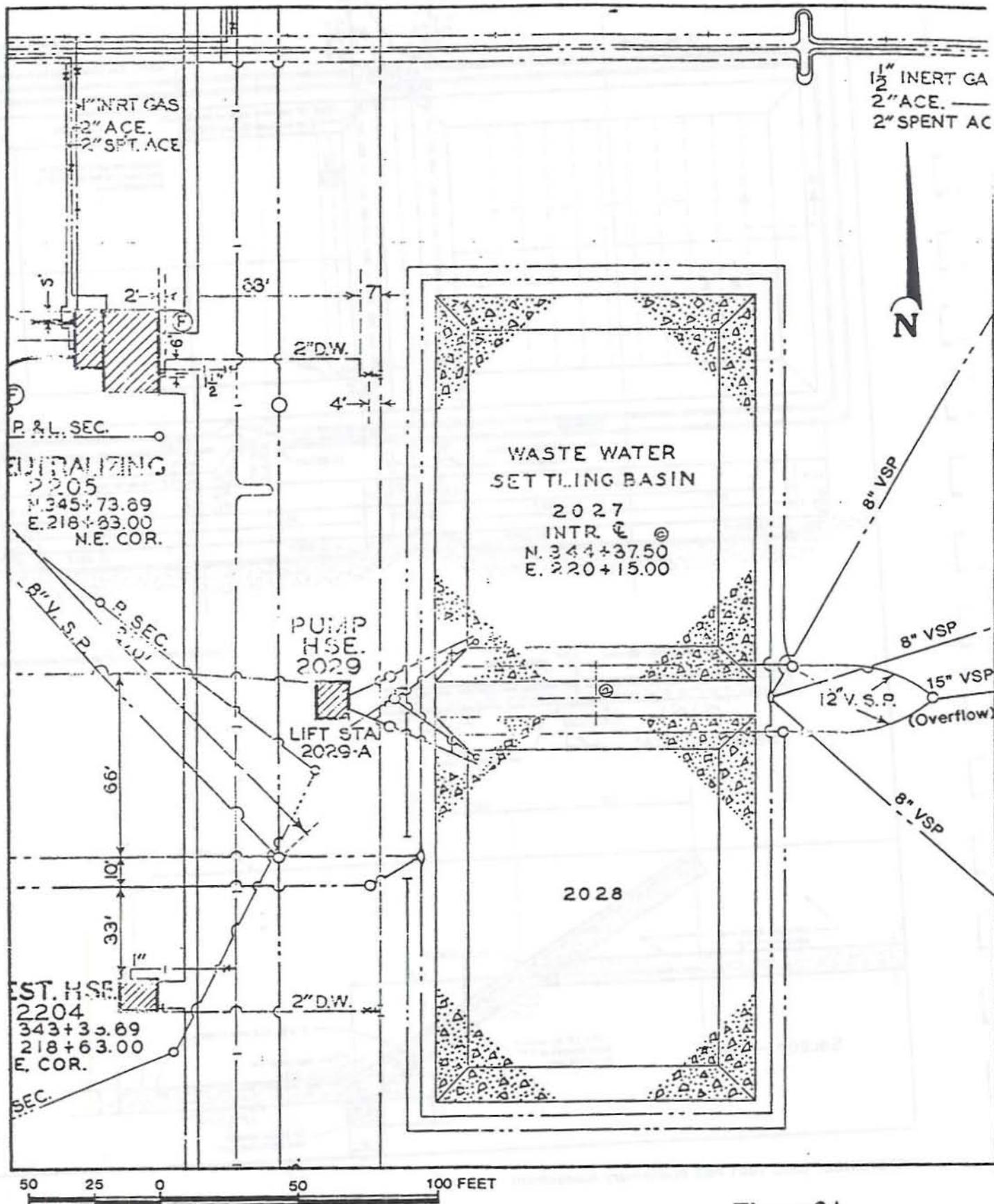
Design By: CRK	Drawn By: WDB	Checked By:	 US Army Corps of Engineers	SITE LOCATION PLAN PENTOLITE AREA WASTE LAGOONS PLUM BROOK ORDNANCE WORKS SANDUSKY, OHIO	
Reviewed By:	Approved By: CRK				
Date: AUG 1999	Scale: NONE	Drawing Code:	Sheet Ref. No.		

FIGURE 2



Design By:	Drawn By:	Checked By:	 US Army Corps of Engineers	PLUM BROOK STATION SANDUSKY, OHIO LIMITED SITE INVESTIGATION FY 99	PROPOSED SAMPLE LOCATIONS PENTOLITE AREA WASTE LAGOONS
Reviewed By:	Approved By:				
Date: 10 MAY 99	Scale: 1" = 200'	Drawing Code:	Sheet Ref. No.:	Figure 2	

File Plot



KEY:

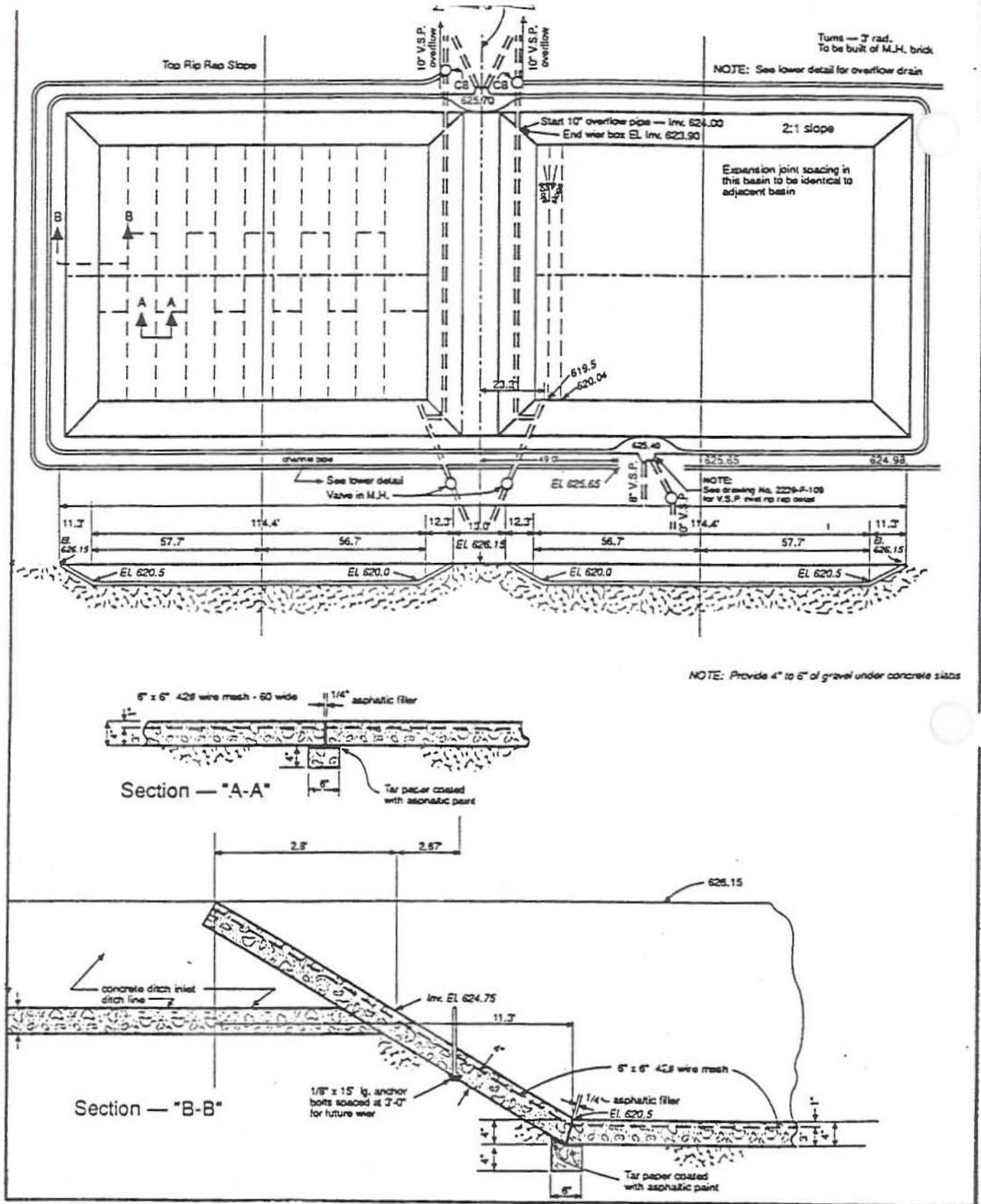
VSP Vitreous Sewer Pipe

BASE: Plan No. R-42, "Unit Layout Map, Pentolite Area",
E.B. Badger & Sons (no date)

Figure 3A

LAYOUT OF
PENTOLITE WASTE WATER BASINS

Plum Brook Ordnance Works
Sandusky, Ohio



REFERENCE: SAIC 1991 PBS Preliminary Assessment

Figure 3B

CONSTRUCTION DETAILS OF
PENTOLITE WASTE WATER BASIN
Plum Brook Ordnance Works
Sandusky, Ohio

APPENDIX A

ETHICS AGREEMENTS

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Deborah A. Hula (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at
(Laboratory): Quanterra, Inc.

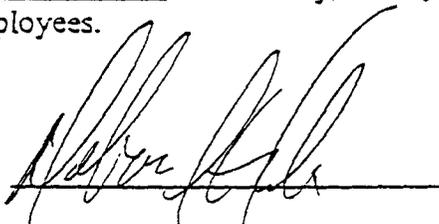
a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)

4/21/99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Spacy D. Campbell (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Spacy D. Campbell

(Signature)

4/20/99

(Date)

Roger

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Roger Toth (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Roger K. Toth

(Signature)

4-21-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Stephen Jackson (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at
(Laboratory): QUANTERRA INC.

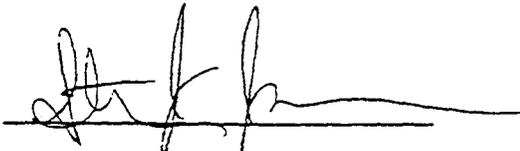
a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)

4-21-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Barbara Girard (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Barbara Girard

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, DORIS HERREN (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Doris Herren

(Signature)

4.20.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Michele Daley (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Michele Daley

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Litty McClain (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Litty McClain
(Signature)

4-20-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Kris M. Buzash (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Kris M Buzash

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Lois Ezzo (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, GREGORY B NEWMAN (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Gregory B Newman

(Signature)

4/29/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Al Haidet (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Amanda K Hercules (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

A K Hercules

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Michael Stamon (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Michael Stamon

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Opal Davis-Johnson (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

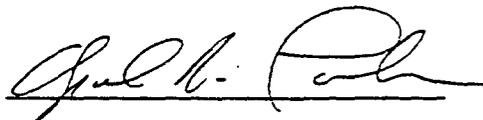
I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Brenda R. Pettay-Kravetz (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Brenda R. Pettay-Kravetz

(Signature)

04/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Dorothy J. Leeson (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)


(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Luis A. Huerta (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Luis A. Huerta
(Signature)

4/20/99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, David H. Cox (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

David H. Cox

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Melissa A. Fuller-Gustave (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Melissa A. Fuller-Gustave
(Signature)

20 April 1999
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Julie Kuhle (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): QUANTERRA, Inc.

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Julie A. Kuhle
(Signature)

990420
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, Susan Macenczak (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).
- II. I agree that in the performance of my duties at Quanterra, Inc. (Laboratory):
- I shall not intentionally report data values that are not the actual values obtained;
 - I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - I shall not intentionally represent another individual's work as my own.
- III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.
- IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Susan A Macenczak

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Tanesha A. Roberts (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- Quanterra
- a. I shall not intentionally report data values that are not the actual values obtained;
 - b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Tanesha A. Roberts
(Signature)

4-20-09
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Laurel Wood (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Laurel E. Wood

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Karen Counts (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Karen L. Counts

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

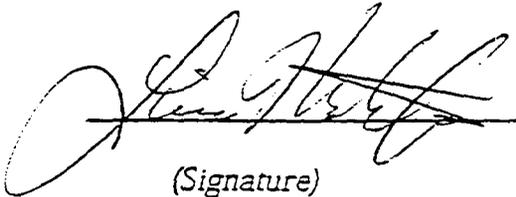
I, LINA Mustafa (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, Melissa Cordell (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).
- II. I agree that in the performance of my duties at (Laboratory):
- a. I shall not intentionally report data values that are not the actual values obtained;
 - b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - c. I shall not intentionally represent another individual's work as my own.
- III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.
- IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Melissa Cordell

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, Heather M. Bosworth (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).
- II. I agree that in the performance of my duties at (Laboratory):
- I shall not intentionally report data values that are not the actual values obtained;
 - I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - I shall not intentionally represent another individual's work as my own.
- III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.
- IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Heather M. Bosworth

(Signature)

4.20.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Scott Zouma (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Scott Zouma

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Albert D DiPofi (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Albert D DiPofi

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, LARRY R. Williams (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Larry R. Williams

(Signature)

4-20-1999

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Lisa McGall (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Lisa McGall

(Signature)

4.21.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, BRYAN M. GREENWELL (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Bryan M. Greenwell
(Signature)

4/20/99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Kim O. Davis (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Kim O. Davis

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Caryne Raach (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Caryne Raach
(Signature)

4-20-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Carolyn Van Doren (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Carolyn S. Van Doren

(Signature)

4.20.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Paul A. Sharkey (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Paul A. Sharkey

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Susan Dempster (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Susan Dempster

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Rhonda Kuster (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra, Inc.

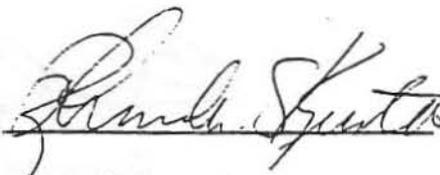
a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, Bradley Bedding (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).
- II. I agree that in the performance of my duties at (Laboratory):
- I shall not intentionally report data values that are not the actual values obtained;
 - I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - I shall not intentionally represent another individual's work as my own.
- III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.
- IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Bradley E. Bedding
(Signature)

4/2/99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, DAVID S. HEAKIN (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

David S. Heakin
(Signature)

4/20/99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, DENISE J. POHL (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Denise J. Pohl

(Signature)

April 20, 1999

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, J. P. C. Smith (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

J. P. C. Smith
(Signature)

April 20, 1999
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Kenneth Kuzior (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Kenneth Kuzior

(Signature)

4/26/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

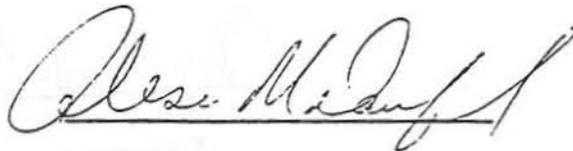
I, Alesia Danford (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Amy McCormick (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Amy McCormick
(Signature)

4-20-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Gary L. Wood (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

April 20, 1999

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, REBECCA L. STRAIT (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Rebecca L. Strait

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Tom Hunt (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Tom Hunt

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, MARK ULMAN (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Mark Ullman

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, John Gruber (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

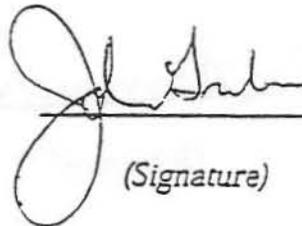
a. I shall not intentionally report data values that are not the actual values obtained;

I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4-26-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Tammy Tokos (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

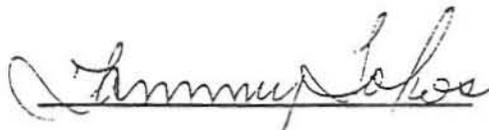
a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)

4.20.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, COOPER WALSH (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

C Walsh
(Signature)

4-21-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Eric S Miller (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Eric S Miller

(Signature)

4/21/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Thomas R Faught (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Thomas R Faught

(Signature)

4/21/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, Donna Williamson (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).
- II. I agree that in the performance of my duties at (Laboratory):
- a. I shall not intentionally report data values that are not the actual values obtained;
 - b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - c. I shall not intentionally represent another individual's work as my own.
- III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.
- IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Donna Williamson

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, IFE NELMES (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, LOUIS MANCINI (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).
- II. I agree that in the performance of my duties at (Laboratory):
- I shall not intentionally report data values that are not the actual values obtained;
 - I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - I shall not intentionally represent another individual's work as my own.
- III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.
- IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Louis Mancini

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Sharon Beis (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Sharon Beis

(Signature)

4-21-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Kami Rathburn (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4/21/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Nathan Pietras (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Nathan Pietras

(Signature)

4-21-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, JON W. RITTENDOR (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)

4-21-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Chris Ciprandi (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at Quanterra Inc. (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Chris Ciprandi
(Signature)

4-20-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Laura Evans (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at Quanterra, North Canton, (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Laura Evans

(Signature)

4/20/99

(Date)

Appendix E

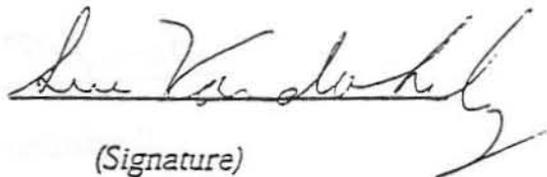
Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, Sue Kardoholy (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).
- II. I agree that in the performance of my duties at (Laboratory):
- a. I shall not intentionally report data values that are not the actual values obtained;
 - b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
 - c. I shall not intentionally represent another individual's work as my own.
- III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.
- IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Kevin M Daniel (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Kevin M Daniel

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Mike Horvath (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Mike Horvath

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

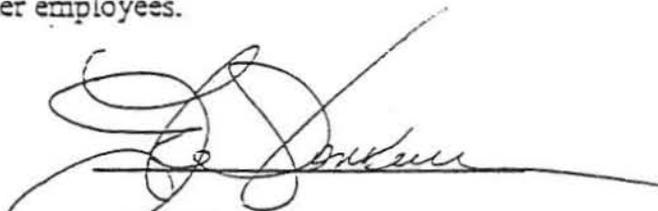
I, LESLIE M. VAN KUREN (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.


(Signature)

990420

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Angel Siegfried (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Angel J. Siegfried
(Signature)

4/20/99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, JENNIFER C. McCRAW (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Jennifer C. McCraw
(Signature)

4-20-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Thomas E. Stiller (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

.. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Thomas E. Stiller

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, William R Cordell (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

William R Cordell

(Signature)

4/21/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, JOSEPH D. GRANT (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Jennifer A. Girard (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Jennifer A. Girard
(Signature)

4-20-99
(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Jami Stephens (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): Quanterra

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Jami Stephens

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

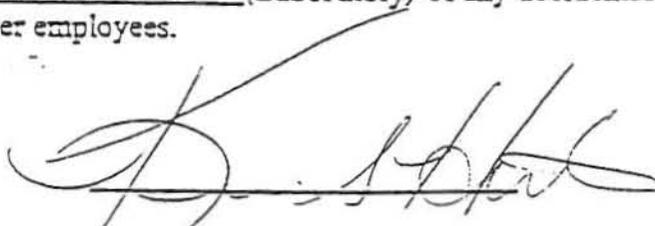
I, Kim Hutras (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory): QUANTERRA INC.

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4/21/99

(Date)

Riley

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, Riley L. Salmons (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Riley L. Salmons

(Signature)

4-21-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

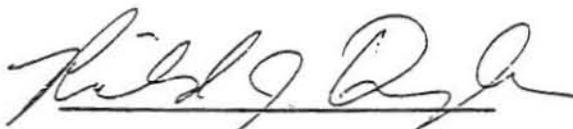
I, RICHARD J. QUAYLE (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.



(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I, SHERRYL ARMAN (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

a. I shall not intentionally report data values that are not the actual values obtained;

b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Sherryl Arman

(Signature)

4.20.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Mark Bruce (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Quanterra, Inc. (Laboratory).

II. I agree that in the performance of my duties at (Laboratory):

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
- c. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Quanterra, Inc. (Laboratory) of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform Quanterra, Inc. (Laboratory) of any accidental or intentional reporting of non-authentic data by other employees.

Mark Bruce

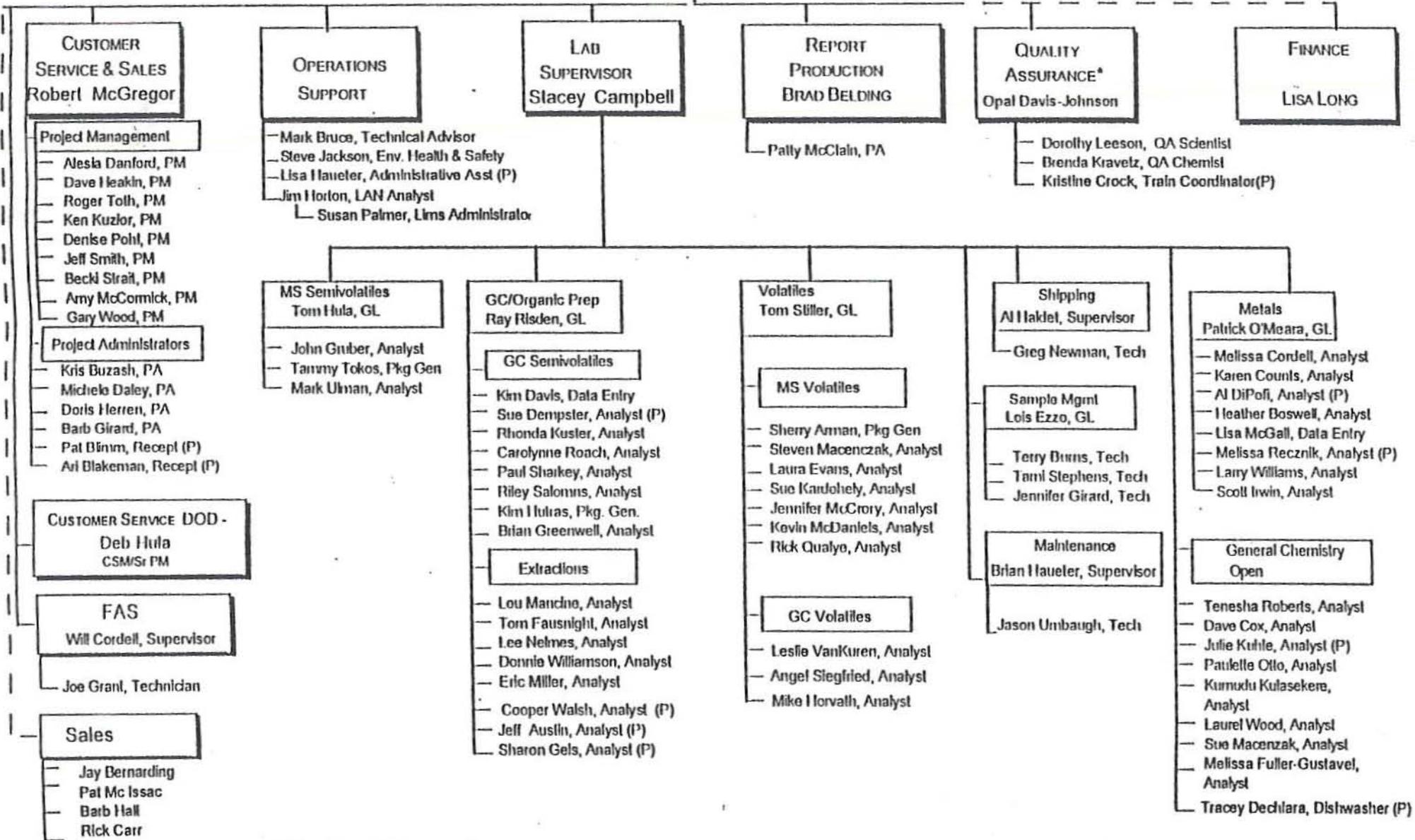
(Signature)

4/20/99

(Date)

QUANTERRA - NORTH CANTON

**LABORATORY
DIRECTOR
Chris Oprandi**



Full-Time Employees 78
 Hourly Employees (P) 12
 TOTAL 90

GL - Group Leader

UNCONTROLLED COPY

Controlled Copy
Copy No: _____

SOP No: CORP-MS-0001NC
Revision No: 1.3
Revision Date: 05/09/97
Page 1 of 52

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHOD 8270B, SW-846

(SUPERSEDES: CORP-MS-0001 (Revision 1.2))

Prepared by: Tom Hux

Reviewed by: John M. Dunbar
Technology Specialist

Approved by: Pauline Cole 5/12/97
Quality Assurance Manager

Approved by: [Signature] 5-12-97
Environmental Health and Safety Coordinator

Approved by: Christopher R. Opando 5-12-97
Laboratory Director

Approved by: [Signature]
Corporate Quality Assurance or Technology

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend or otherwise dispose or disclose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

1. SCOPE AND APPLICATION	4
2 SUMMARY OF METHOD	5
3 DEFINITIONS	5
4 INTERFERENCES	6
5 SAFETY PRECAUTIONS	6
6 EQUIPMENT AND SUPPLIES	7
7 REAGENTS AND STANDARDS	8
8 SAMPLE PRESERVATION AND STORAGE	9
9 QUALITY CONTROL	9
10 CALIBRATION AND STANDARDIZATION	14
11 PROCEDURE	18
12 DATA ANALYSIS AND CALCULATIONS	20
13 METHOD PERFORMANCE	26
14 POLLUTION PREVENTION	28
15 WASTE MANAGEMENT	28
16 REFERENCES	28
17 MISCELLANEOUS	28
18. REQUIREMENTS FOR METHOD 625	51

LIST OF TABLES

TABLE 1	Quanterra Primary Standard and Standard Reporting Limits
TABLE 2	Quanterra Appendix IX Standard and Standard Reporting Limits
TABLE 3	Reportable Analytes for Quanterra Standard Tests, Primary Standard
TABLE 4	Reportable analytes for Quanterra Standard Tests, Appendix IX Standard
TABLE 5	Recommended Instrument Conditions
TABLE 6	DFTPP Ion Abundance Criteria
TABLE 7	Characteristic Ions, Primary Standard
TABLE 8	Characteristic Ions, Appendix IX Standard
TABLE 9	8270B LCS Compounds
TABLE 10	TCLP LCS Compounds
TABLE 11	8270B Surrogate Compounds
TABLE 12	Calibration Levels, Primary Standard
TABLE 13	Calibration Levels, Appendix IX Standard
TABLE 14	Initial Demonstration Accuracy and Precision Limits
TABLE A-1	Method 625 Reporting List and Limits
TABLE-A-2	Method 625 LCS and MS Compounds and Spike Concentrations

1. SCOPE AND APPLICATION

- 1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.
- 1.2 The following compounds may require special treatment when being determined by this method:
- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
 - Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
 - N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
 - Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
 - Hexachlorophene is not amenable to analysis by this method.
 - 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

- 1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2 SUMMARY OF METHOD

- 2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel, a continuous extractor or Accelerated One-Step™. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or pressurized fluid extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3 DEFINITIONS

- 3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.
- 3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.
- 3.3 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. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent 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.
- 3.4 Method Blank - An analytical control consisting of all reagents, 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 and reagent

contamination.

- 3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.
- 3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.
- 3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4 INTERFERENCES

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.
- 4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.
- 4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.
- 4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.
- 4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

- 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:
- 5.1.1 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 become contaminated will be removed and discarded; other gloves will be cleaned immediately.
 - 5.1.2 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:
 - 5.1.3 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.
 - 5.1.4 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. Solvent and waste containers should be kept closed unless transfers are being made.
 - 5.1.5 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.

6 EQUIPMENT AND SUPPLIES

- 6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.
- 6.2 Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5- μ m film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.
- 6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or

less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS tuning standard is injected through the GC.

- 6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.
- 6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.
- 6.6 Syringe: 10 μ L Hamilton Laboratory grade syringes or equivalent.
- 6.7 Carrier gas: Ultra high purity helium.

7 REAGENTS AND STANDARDS

- 7.1 A minimum five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.
- 7.2 An Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.
 - 7.2.1 Internal Standards are added to all standards and extracts to result in 8 ng or 40ng injected onto the column. For example, if the volume of an extract used was 200 μ L, 20 μ L of a 400 μ g/mL internal standard solution would be added for a 1 μ L injection.
- 7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.
- 7.4 GC/MS Tuning Standard: A methylene chloride solution containing 10 μ g/mL or 50

$\mu\text{g/mL}$ of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at $10 \mu\text{g/mL}$ or $50 \mu\text{g/mL}$.

- 7.5 Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.
- 7.6 Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.
- 7.7 The standards listed in 7.1 to 7.6 should be refrigerated at $\leq 6^{\circ}\text{C}$ when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at $\leq 6^{\circ}\text{C}$.

8 SAMPLE PRESERVATION AND STORAGE

- 8.1 Reference appropriate facility SOP for sample bottle preservation and storage.
- 8.2 Samples are stored at $4 \pm 2^{\circ}\text{C}$. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)
- 8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9 QUALITY CONTROL

- 9.1 Initial Demonstration of Capability
 - 9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. Refer to the flow chart in section 17.4.1.
 - 9.1.2 For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2 Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery \pm 3 standard deviations for surrogates, MS and LCS. Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference \pm 3 standard deviations.

- 9.2.1 These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.
- 9.2.2 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.
- 9.2.3 Refer to the QC program document (QA-003) for further details of control limits.

9.3 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (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 (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.
- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

- 9.3.1 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in

the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.2 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.3 Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.3.4 Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4 Instrument Blank

9.4.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency.

9.5.2 If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to

provide further documentation.

9.5.3 Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2 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.
- Re-extract and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem.

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.

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

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

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1 Inject 10 ng or 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be $< 20\%$. Refer to section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor

response. Add the internal standard mixture to result in 8 ng or 40 ng on column. The concentrations of all analytes are listed in tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene

Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the average of all %RSDs in the initial calibration is $\leq 15\%$, then all analytes may use average response factor for calibration.

10.4.7.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD $> 15\%$ for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.4.7.2 If the average of all the %RSDs in the initial calibration is $> 15\%$, then calibration on a curve must be used for those analytes with %RSD $> 15\%$. Linear or quadratic curve fits may be used. Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990 . If the correlation coefficient is < 0.990 , then any hit for these compounds must be flagged as estimated.

10.4.8 Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.9 If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.10 Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.5 Continuing Calibration

10.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 10 ng or 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.5.3 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05 .
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq 20\%$. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq 50\%$, with allowance being made for up to six target compounds to have percent drift greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- NOTE: There is no internal standard criteria for samples. Criteria is only for continuing and initial calibrations.

10.5.3.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

NOTE: Additional compound analysis outside the TCL. (Target Compound List) have no calibration criteria.

11 PROCEDURE

11.1 Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2 Sample Analysis Procedure

- 11.2.1 Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.
- 11.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.
- 11.2.3 Add internal standard to the extract to result in 40 ng injected on column (for example, 8 μ L or 50 μ L internal standard solution in 0.5 mL of extract for a 1 μ L injection). Mix thoroughly before injection into the instrument.
- 11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.
- 11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.
- 11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.
- 11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.
- 11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits

below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 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.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at $4 \pm 2^{\circ}\text{C}$, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 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 the facility specific SOP for determination of percent moisture.

11.7 Procedural Variations

11.7.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix.

radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using 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 also be documented as a non-conformance, with a cause and corrective action described.

11.8 Troubleshooting Guide

11.8.1 Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the Quanterra QAMP, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

11.8.2 Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of

the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- The relative intensities of ions should agree to within $\pm 30\%$ between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1 Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions $>10\%$ of the most abundant ion) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within $\pm 20\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine

Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100$$

RF = Mean of RFs from initial calibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\frac{\sum_{i=1}^N (RF_i - \overline{RF})^2}{N-1}}$$

RF_i = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is $\leq 15\%$, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} RF}$$

12.5.3.2 Linear fit

$$C_{ex} = A + B \frac{(R_x C_{is})}{R_{is}}$$

C_{ex} = Concentration in extract, $\mu\text{g/mL}$

R_x = Response for analyte

R_{is} = Response for internal standard

C_{is} = Concentration of internal standard

A = Intercept

B = Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B \left(\frac{R_x C_{is}}{R_{is}} \right) + C \left(\frac{R_x C_{is}}{R_{is}} \right)^2$$

C = Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu\text{g/L} = \frac{C_{ex} V_i}{V_o}$$

Where:

V_i = Volume of total extract, μL , taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean $V_i = 10.000$)

μL . If half of the base/neutral extract and half of the acid extract are combined, $V_t = 2,000$.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis):

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_e V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_x = Concentration of the compound being measured ($\mu\text{g/L}$)

C_{is} = Concentration of the specific internal standard ($\mu\text{g/L}$)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

$$RF = 1$$

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

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 an initial demonstration of capability for each individual

method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

13.2.3 If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

- 13.5 Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14 POLLUTION PREVENTION

- 14.1 This section is not applicable to this procedure.

15 WASTE MANAGEMENT

- 15.1 Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16 REFERENCES

- 16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270B.
- 16.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17 MISCELLANEOUS

17.1 Modifications from Reference Method

- 17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.
- 17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Modifications from Previous Revision

- 17.2.1 This SOP has been substantially revised to meet the requirements of method 8270C.
- 17.2.2 Directions for analysis by method 625 have been added as an attachment.
- 17.2.3 SOP has been revised to include a 5 mL final volume.

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 No.	Table Title	Table Description	Table Location
17.4.1	Table 1	Table 1	Table 1
17.4.2	Table 2	Table 2	Table 2
17.4.3	Table 3	Table 3	Table 3
17.4.4	Table 4	Table 4	Table 4
17.4.5	Table 5	Table 5	Table 5
17.4.6	Table 6	Table 6	Table 6
17.4.7	Table 7	Table 7	Table 7
17.4.8	Table 8	Table 8	Table 8
17.4.9	Table 9	Table 9	Table 9
17.4.10	Table 10	Table 10	Table 10
17.4.11	Table 11	Table 11	Table 11
17.4.12	Table 12	Table 12	Table 12
17.4.13	Table 13	Table 13	Table 13
17.4.14	Table 14	Table 14	Table 14
17.4.15	Table 15	Table 15	Table 15
17.4.16	Table 16	Table 16	Table 16
17.4.17	Table 17	Table 17	Table 17
17.4.18	Table 18	Table 18	Table 18
17.4.19	Table 19	Table 19	Table 19
17.4.20	Table 20	Table 20	Table 20
17.4.21	Table 21	Table 21	Table 21
17.4.22	Table 22	Table 22	Table 22
17.4.23	Table 23	Table 23	Table 23
17.4.24	Table 24	Table 24	Table 24
17.4.25	Table 25	Table 25	Table 25
17.4.26	Table 26	Table 26	Table 26
17.4.27	Table 27	Table 27	Table 27
17.4.28	Table 28	Table 28	Table 28
17.4.29	Table 29	Table 29	Table 29
17.4.30	Table 30	Table 30	Table 30
17.4.31	Table 31	Table 31	Table 31
17.4.32	Table 32	Table 32	Table 32
17.4.33	Table 33	Table 33	Table 33
17.4.34	Table 34	Table 34	Table 34
17.4.35	Table 35	Table 35	Table 35
17.4.36	Table 36	Table 36	Table 36
17.4.37	Table 37	Table 37	Table 37
17.4.38	Table 38	Table 38	Table 38
17.4.39	Table 39	Table 39	Table 39
17.4.40	Table 40	Table 40	Table 40
17.4.41	Table 41	Table 41	Table 41
17.4.42	Table 42	Table 42	Table 42
17.4.43	Table 43	Table 43	Table 43
17.4.44	Table 44	Table 44	Table 44
17.4.45	Table 45	Table 45	Table 45
17.4.46	Table 46	Table 46	Table 46
17.4.47	Table 47	Table 47	Table 47
17.4.48	Table 48	Table 48	Table 48
17.4.49	Table 49	Table 49	Table 49
17.4.50	Table 50	Table 50	Table 50

Table 1

Quanterra Primary Standard¹ and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane) ²	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330

Table 1

Quanterra Primary Standard¹ and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

¹ The Quanterra primary standard is the standard normally used at Quanterra. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

² 2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2

Quanterra Appendix IX¹ Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate ²	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin ²	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp ²	3689-24-5	50	1600
Phorate ²	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate ³	2303-16-4	20	660
Dimethoate ²	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton ²	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
*Methyl Parathion ²	298-00-0	50	1600

Table 2

Quanterra Appendix IX¹ Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4-Nitroquinoline-1-oxide	56-57-5	100	3300
*Parathion ²	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
*Isodrin ³	465-73-6	10	330
*Kepone ²	143-50-0	100	3300
Famphur ³	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate ³	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

- ¹ The Appendix IX standard contains additional analytes required for the Appendix IX list. The Quanterra primary standard must also be analyzed to include all of the Appendix IX list.
- ² May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.
- ³ It is highly recommended that Famphur is analyzed by method 8081. It is a poor responder by 8270C.
- * These compounds are analyzed by GC methods at Quanterra, North Canton.

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane) ¹	180-60-1	X		X	X
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethyl phthalate	84-66-2	X		X	X

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene ¹	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Buryl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X

¹ 2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

² Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2				X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
3-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate ²	126-68-1				X
a,a-Dimethyl-phenethylamine	122-09-8				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				X
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin ²	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp ²	3689-24-5				X
Phorate ²	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate ²	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton ²	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb) ²	88-85-7				X
*Methyl parathion ²	298-00-0				X

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
4-Nitroquinoline-1-oxide	56-57-5				X
*Parathion ²	56-38-2				X
*Isodrin ³	465-73-6				X
*Kepone ²	143-50-0				X
Famphur ²	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate ³	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene ⁴	70-30-4				X
Diphenylamine ⁵	122-39-4				X

² May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

³ May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

⁴ Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See section 12.2.1)

⁵ Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

* These compounds are analyzed by GC methods at Quanterra, North Canton.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	<1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	1 or 2 μ l
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/ μ L in extract ¹
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

¹ Levels are 50 and 75 ng/ μ L if 2 μ L injection is used

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/ μ L in extract ¹
1,4-Dichlorobenzene	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

¹ Levels are 50 ng/ μ L if 2 μ L injection is used

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/ μ L in extract ²
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4 ¹	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4 ¹	150

¹ Included in standard mix, but not routinely evaluated for method 8270B

² Levels are 50 and 75 ng/ μ L if 2 μ L injection is used

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12³
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Pyridine	10	25	40	60	80
N-nitrosodimethylamine	10	25	40	60	80
Aniline	10	25	40	60	80
Phenol	10	25	40	60	80
Bis(2-chloroethyl)ether	10	25	40	60	80
2-Chlorophenol	10	25	40	60	80
1,3-Dichlorobenzene	10	25	40	60	80
1,4-Dichlorobenzene	10	25	40	60	80
Benzyl alcohol	10	25	40	60	80
1,2-Dichlorobenzene	10	25	40	60	80
2-Methylphenol	10	25	40	60	80
2,2'-oxybis(1-chloropropane) ¹	10	25	40	60	80
4-Methylphenol	10	25	40	60	80
N-Nitroso-di-n-propylamine	10	25	40	60	80
Hexachloroethane	10	25	40	60	80
Nitrobenzene	10	25	40	60	80
Isophorone	10	25	40	60	80
2-Nitrophenol	10	25	40	60	80
2,4-Dimethylphenol	10	25	40	60	80
Benzoic acid	10	25	40	60	80
Bis(2-chloroethoxy)methane	10	25	40	60	80
2,4-Dichlorophenol	10	25	40	60	80
1,2,4-Trichlorobenzene	10	25	40	60	80
Naphthalene	10	25	40	60	80
4-Chloroaniline	10	25	40	60	80
Hexachlorobutadiene	10	25	40	60	80
4-Chloro-3-methylphenol	10	25	40	60	80
2-Methylnaphthalene	10	25	40	60	80
Hexachlorocyclopentadiene	10	25	40	60	80
2,4,6-Trichlorophenol	10	25	40	60	80
2,4,5-Trichlorophenol	10	25	40	60	80
2-Chloronaphthalene	10	25	40	60	80
2-Nitroaniline	10	25	40	60	80
Dimethyl phthalate	10	25	40	60	80
Acenaphthylene	10	25	40	60	80
3-Nitroaniline	10	25	40	60	80
Acenaphthene	10	25	40	60	80
2,4-Dinitrophenol	10	25	40	60	80
4-Nitrophenol	10	25	40	60	80
Dibenzofuran	10	25	40	60	80
2,4-Dinitrotoluene	10	25	40	60	80
2,6-Dinitrotoluene	10	25	40	60	80
Diethyl phthalate	10	25	40	60	80
4-Chlorophenyl phenyl ether	10	25	40	60	80
Fluorene	10	25	40	60	80
4-Nitroaniline	10	25	40	60	80

Table 12³
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
4,6-Dinitro-2-methylphenol	10	25	40	60	80
N-Nitrosodiphenylamine	10	25	40	60	80
Azobenzene ²	10	25	40	60	80
4-Bromophenyl phenyl ether	10	25	40	60	80
Hexachlorobenzene	10	25	40	60	80
Pentachlorophenol	10	25	40	60	80
Phenanthrene	10	25	40	60	80
Anthracene	10	25	40	60	80
Carbazole	10	25	40	60	80
Di-n-butyl phthalate	10	25	40	60	80
Fluoranthene	10	25	40	60	80
Benzidine	10	25	40	60	80
Pyrene	10	25	40	60	80
Butyl benzyl phthalate	10	25	40	60	80
3,3'-Dichlorobenzidine	10	25	40	60	80
Benzo(a)anthracene	10	25	40	60	80
Bis(2-ethylhexyl)phthalate	10	25	40	60	80
Chrysene	10	25	40	60	80
Di-n-octylphthalate	10	25	40	60	80
Benzo(b)fluoranthene	10	25	40	60	80
Benzo(k)fluoranthene	10	25	40	60	80
Benzo(a)pyrene	10	25	40	60	80
Indeno(1,2,3-cd)pyrene	10	25	40	60	80
Dibenz(a,h)anthracene	10	25	40	60	80
Benzo(g,h,i)perylene	10	25	40	60	80

¹ 2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

² Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

³ Optional concentration levels = 1/5 of the listed concentrations for samples with a final volume of 5 mL.

Table 13¹

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	25	40	60	80
N-Nitrosomethylethylamine	10	25	40	60	80
Methyl methanesulfonate	10	25	40	60	80
N-Nitrosodiethylamine	10	25	40	60	80
Ethyl methanesulfonate	10	25	40	60	80
Pentachloroethane	10	25	40	60	80
Acetophenone	10	25	40	60	80
N-Nitrosopyrrolidine	10	25	40	60	80
N-Nitrosomorpholine	10	25	40	60	80
o-Toluidine	10	25	40	60	80
3-Methylphenol	10	25	40	60	80
N-Nitrosopiperidine	10	25	40	60	80
o,o,o-Triethyl-Phosphorothioate	10	25	40	60	80
a,a-Dimethyl-phenethylamine	10	25	40	60	80
2,6-Dichlorophenol	10	25	40	60	80
Hexachloropropene	10	25	40	60	80
p-Phenylenediamine	10	25	40	60	80
n-Nitrosodi-n-butylamine	10	25	40	60	80
Safrole	10	25	40	60	80
1,2,4,5-Tetrachlorobenzene	10	25	40	60	80
Isosafrole 1 + 2	10	25	40	60	80
1,4-Dinitrobenzene	10	25	40	60	80
1,4-Naphthoquinone	10	25	40	60	80
1,3-Dinitrobenzene	10	25	40	60	80
Pentachlorobenzene	10	25	40	60	80
1-Naphthylamine	10	25	40	60	80
2-Naphthylamine	10	25	40	60	80
2,3,4,6-Tetrachlorophenol	10	25	40	60	80
5-Nitro-o-toluidine	10	25	40	60	80
Thionazin	10	25	40	60	80
1,3,5-Trinitrobenzene	10	25	40	60	80
Sulfotepp	10	25	40	60	80
Phorate	10	25	40	60	80
Phenacetin	10	25	40	60	80
Diallate 1 + 2	10	25	40	60	80
Dimethoate	10	25	40	60	80
4-Aminobiphenyl	10	25	40	60	80
Pentachloronitrobenzene	10	25	40	60	80
Pronamide	10	25	40	60	80
Disulfoton	10	25	40	60	80
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	25	40	60	80
*Methyl parathion	10	25	40	60	80
4-Nitroquinoline-1-oxide	10	25	40	60	80
*Parathion	10	25	40	60	80

Table 13¹

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
*Isodrin	10	25	40	60	80
*Kepone	10	25	40	60	80
Famphur	10	25	40	60	80
Methapyrilene	10	25	40	60	80
Aramite 1 and 2	10	25	40	60	80
p-(Dimethylamino)azobenzene	10	25	40	60	80
p-Chlorobenzilate	10	25	40	60	80
3,3'-Dimethylbenzidine	10	25	40	60	80
2-Acetylaminofluorene	10	25	40	60	80
Dibenz (a,j)acridine	10	25	40	60	80
7,12-Dimethylbenz(a)anthracene	10	25	40	60	80
3-Methylcholanthrene	10	25	40	60	80

* These compounds are not in the Quanterra North Canton Standard Mix,

²Option: concentration levels = 1/5 of the listed concentrations for samples with a final volume of 5 mL.

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin ¹	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benzo(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC ¹	60	31.5	41.5-130.6
d-BHC ¹	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-112.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
1,1'-DDD ¹	60	31.0	D-134.5

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
4,4'-DDE ¹	60	32.0	19.2-119.7
4,4'-DDT ¹	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin ¹	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate ¹	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor ¹	60	37.2	D-172.2
Heptachlor epoxide ¹	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260 ¹	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration $\mu\text{g/L}$	Limit for Relative Standard Deviation	Limit for average recovery, %
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

¹Since the organochlorine pesticides and PCBs are normally determined by method 8080 at Quanterra, they will not be included in the initial demonstration of capability for method 8270B.

ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING
METHOD 625

18. REQUIREMENTS FOR METHOD 625

- 18.1 Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.
- 18.2 This method can be applied only to aqueous matrices.
- 18.3 The tune period for this method is defined as 24 hours.
- 18.4 Initial calibration curve requirements:
- 18.4.1 The initial calibration curve for this method requires at least three points.
 - 18.4.2 Target compounds must have $RSD \leq 35\%$.
 - 18.4.3 If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.
- 18.5 Continuing calibration verification requirements: All target compounds must have $\%D \leq 20\%$.
- 18.6 Matrix Spike and LCS requirements:
- 18.6.1 A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1. Quanterra Method 625 standard reporting list and reporting limits.

Analytes	CAS Number	Aqueous
		µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10

Analytes	CAS Number	Aqueous
		µg/L
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2. Method 625 LCS and MS compounds and spike concentrations.

LCS Compounds	Spiking Level, ng/ μ L in extract ¹
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100

LCS Compounds	Spiking Level, ng/ μ L in extract ¹
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

¹ Levels are 50 and 75 ng/ μ L if 2 μ L injection is used

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: AQUEOUS ICP & FLAA ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS

(SUPERSEDES: ENSECO SOP #LM-WALN-2520 (REVISION 1.0))

Prepared by: Susan J Palmer

Reviewed by: Brenda Repasky
Technology Specialist

Approved by: Paul H. Cohen
Quality Assurance Manager

Approved by: Shirley A. Pitt
Environmental Health and Safety Coordinator

Approved by: Chris Opinski for Robert George
Laboratory Director

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held propriety to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any propose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

1. SCOPE AND APPLICATION3

2. SUMMARY OF METHOD3

3. DEFINITIONS3

4. INTERFERENCES3

5. SAFETY3

6. EQUIPMENT AND SUPPLIES4

7. REAGENTS AND STANDARDS5

8. SAMPLE COLLECTION, PRESENTATION AND STORAGE6

9. QUALITY CONTROL6

10. CALIBRATION AND STANDARDIZATION7

11. PROCEDURE7

12. DATA ANALYSIS AND CALCULATIONS9

13. METHOD PERFORMANCE9

14. POLLUTION PREVENTION10

15. WASTE MANAGEMENT10

16. REFERENCES10

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)11

1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the acid digestion of aqueous samples, EP and mobility procedure extracts, and wastes containing suspended solids for analysis by ICP or FLAA of all metals except Mercury. This method is based on EPA Method 200.7 and SW846 Method 3010A and 3005A for the determination of total and recoverable metals.
- 1.2. This digestion procedure is not suitable for samples which will be analyzed by Graphite Furnace Atomic Absorption Spectroscopy, because hydrochloric acid can cause interferences during furnace atomization.
- 1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

- 2.1. A mixture of HNO₃, HCl and sample to be analyzed is refluxed in a beaker. The sample volume is reduced and diluted to volume.
- 2.2. For dissolved metals, at the time of collection, the sample is filtered through a 0.45 µm filter and the liquid phase is then acidified with nitric acid. Samples for dissolved metals do not need to be digested as long as the acid concentrations have been adjusted to the same concentration as in the standards.

3. DEFINITIONS

- 3.1. Refer to the glossary in 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 that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

- 5.1. 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. The following specific hazards are known:
- 5.3.1. The following materials are known to be **corrosive: Hydrochloric Acid, Nitric Acid**
- 5.4. 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.
- 5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. Beakers: 100 mL or 150 mL
- 6.2. Watchglasses
- 6.3. Filter Paper: qualitative grade
- 6.4. Hotplate: capable of maintaining $95^{\circ}\text{C} \pm 4^{\circ}\text{C}$. or equivalent heating source
- 6.5. Calibrated Thermometer: $0^{\circ}\text{C} - 200^{\circ}\text{C}$. used to check hotplate temperature
- 6.6. Volumetric Flasks: 50 mL. class A
- 6.7. Graduated Cylinders or equivalent: various sizes

- 6.8. Funnels
- 6.9. Corning Graduated Plastic Bottles or equivalent
- 6.10. Dispenser
- 6.11. Reagent Water Squirt Bottle
- 6.12. 1:1 HCl Squirt Bottle
- 6.13. Eppendorf and Tips

7. REAGENTS AND STANDARDS

7.1. Reagents

- 7.1.1. Nitric Acid (HNO₃), Concentrated: Tracepur Plus
- 7.1.2. Hydrochloric Acid (HCl), Concentrated: Tracepur Plus
- 7.1.3. Reagent Water
- 7.1.4. Hydrochloric Acid (1:1): Add 500 mL of concentrated HCl to 500 mL of Reagent Water

7.2. Standards

7.2.1. Primary Stock Standards

7.2.1.1. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source. Standards must have a purity of 99.5% or greater. Standards may be purchased with individual elements or purchased in mixes.

7.2.1.2. Stock standards solutions must be replaced after one year or sooner if comparison with a second source standard indicates a problem. Standard solutions should be checked frequently for signs of degradation or evaporation.

7.2.2. Laboratory Control Samples (LCS)

7.2.2.1. From the stock standard prepare a laboratory control sample (LCS) from a source other than that used for the calibration standards at a concentration listed in Table 1.

7.2.3. Matrix Spike Standard

7.2.3.1. From the stock standard prepare a matrix spike standard of known concentration for use as an MS/MSD (Section 9.4) at a concentration listed in Table 1.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. All samples are acidified to pH < 2 with HNO₃ and stored at room temperature in plastic or glass containers with screw caps.
- 8.2. For dissolved metals, all samples must be filtered through 0.45 µm filter paper and then acidified at the time of collection with HNO₃ to a pH of <2. If this procedure is not performed in the field, it must be performed prior to digestion.
- 8.3. The holding time is six months from sampling to completion of analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. 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.

9.3.2. A midrange LCS must be prepared with each batch of samples.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each 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/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. If an MS/MSD is not possible due to limited sample volume, a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS/LCSD must be compared to the matrix spike RPD limits.

9.5. Sample Duplicates

9.5.1. At a client's request, MSs and duplicates can be performed in place of MS/MSDs.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical

Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation Procedure for Total Recoverable Metals (Method 3005A)

11.3.1. Transfer a 50 mL aliquot of a well-mixed sample to a beaker.

NOTE: If it is necessary to use sample volumes other than 50 mL, adjust all other volumes proportionately.

NOTE: LCS and Matrix Spike standards (sections 7.2.2 and 7.2.3) may be added either before or after the addition of acids; however, consistency should be maintained throughout the procedure.

11.3.2. Add 1 mL of concentrated HNO₃ and 5 mL of 1:1 HCl, cover with a watchglass, place the beaker on a hotplate, and reflux at 95°C ±4°C, reducing the volume of sample to approximately 15-20 mL. Do not allow the sample to boil or go to dryness at any stage of the digestion process. If the sample boils or goes to dryness, the sample must be reprepared.

NOTE: Do not boil. Antimony is easily lost by volatilization from HCl media.

11.3.3. Let the beaker cool and remove the watchglass. Transfer the sample to a Corning graduated plastic bottle with snap top. Rinse sample residue from the side of the beaker. Rinse twice with reagent water. Collect the rinses in the plastic bottle. Bring the final volume to 50 mL with reagent water. Securely snap the top on the bottle.

11.3.3.1. If silicates or other insoluble matter are present, filter the sample through qualitative grade filter paper into a Corning graduated plastic bottle with snap top. Rinse the sample's original beaker twice with reagent water. Drain the rinses through the filter. Make a final rinse of the filter with reagent water. Bring the final volume to 50 mL with reagent water. Securely snap the top on the bottle.

11.4. Sample Preparation Procedure for Total Metals (Methods 200.7 and 3010A)

11.4.1. Transfer a 50 mL aliquot of a well-mixed sample to a beaker.

NOTE: If it is necessary to use sample volumes other than 50 mL, adjust all other volumes appropriately.

NOTE: LCS and Matrix Spike standards (sections 7.2.2 and 7.2.3) may be added either before or after the addition of acids; however, consistency should be maintained throughout the procedure.

11.4.2. Using a dispenser, add 3 mL of concentrated HNO₃ and 5 mL of 1:1 HCl. Cover with a watchglass and place on a hotplate. Reflux at 95°C ±4°C, reducing the volume of sample to approximately 20 mL. Do not allow the sample to boil or go to dryness at any stage of the digestion process. If the sample boils or goes to dryness, the sample must be reprepared.

11.4.3. Let the beaker cool and remove the watchglass. Transfer the sample to a Corning graduated plastic bottle with snap top. Rinse the sample residue from the side of the beaker. Rinse twice with reagent water. Collect the rinses in the plastic bottle. Bring the final volume to 50 mL with reagent water. Securely snap the top on the bottle.

11.4.3.1. If silicates or other insoluble matter are present, filter the sample through qualitative grade filter paper into a Corning graduated plastic bottle with snap top. Rinse the sample's original beaker twice with reagent water. Drain the rinses through the filter. Make a final rinse of the filter with reagent water. Bring the final volume to 50 mL with reagent water. Securely snap the top on the bottle.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not Applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. 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 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. Acid waste is disposed of in the sample prep sink with copious amounts of water.
- 15.2. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.
- 15.3. Refer to the Laboratory Sample and Waste Disposal plan.
- 15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Quanterra. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

- 16.1. References
- 16.1.1. SW846, Test Methods for Evaluating Solid Wastes, Third Edition, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, Method 3005A
- 16.1.2. SW846, Test Methods for Evaluating Solid Wastes, Third Edition, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, Method 3010A
- 16.1.3. EPA 200.7, Determination of Total Metals by Inductively Coupled Plasma
- 16.2. Associated SOPs
- 16.2.1. Inductively Coupled Plasma-Atomic Emission Spectroscopy. Spectrometric Method for Trace Elements. CORP-MT-0001
- 16.2.2. Elemental Analysis by Flame Atomic Absorption Spectrophotometry. NC-MT-0001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

SPIKING PROTOCOL

Sample ID	Sample Description	Spiking Solution	Spiking Volume	Final Volume
101	Water	100 ppm Pb	10 mL	100 mL
102	Water	100 ppm Cu	10 mL	100 mL
103	Water	100 ppm Zn	10 mL	100 mL
104	Water	100 ppm Cd	10 mL	100 mL
105	Water	100 ppm Ni	10 mL	100 mL

Table 1: Spiking Protocol

Preparation Type	Matrix	Amt of Std added LCS & MSD	Initial Sample Vol/Wt	Final Sample Volume
ICP	water	1 mL ICP-1 1mL Ag 2.5 mL ICP-2	50 mL	50 mL
Pb, FLAA	water	2.5 mL Pb	50 mL	50 mL
Sn, Ti, FLAA	water	2.5 mL Sn, Ti	50 mL	50 mL

Table 2: Standard Definitions

Elements	Ag	ICP-1	ICP-2	Flame
Ag*	2.5 ppm	2.5 ppm		
Al		100 ppm		
As		100 ppm		
B		50 ppm		
Ba		100 ppm		
Be		2.5 ppm		
Cd		2.5 ppm		
Ca			1000 ppm	
Co		25 ppm		
Cr		10 ppm		
Cu		12.5 ppm		
Fe		50 ppm		
K			1000 ppm	
Mg			1000 ppm	
Mn		25 ppm		
Mo		50 ppm		
Na			1000 ppm	
Ni		25 ppm		
Pb		25 ppm		

**AQUEOUS ICP & FLAA ACID DIGESTION OF
AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS**

SOP No. NC-IP-0003
Revision No. 3
Revision Date: 02/10/99
Page 15 of 16

Elements	Ag	ICP-1	ICP-2	Flame
Sb		25 ppm		
Se		100 ppm		
Sn		100 ppm		
Sr		50 ppm		
Ti		50 ppm		
Tl		100 ppm		
V		25 ppm		
Zn		25 ppm		
Pb FLAA				100 ppm
Sn FLAA				100 ppm
Ti FLAA				100 ppm

*Analysis by ICP only

ODD Elements are prepared separately as needed at the following concentrations:

Bi 100 ppm

Os 100 ppm

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS FOR METAL ANALYSIS BY GFAA, FLAA, OR ICP

(SUPERSEDES: ENSECO SOP #LM-WALN-2530 (REVISION 1.0))

Prepared by: Susan Palmer

Reviewed by: Karen L. Counts
Technology Specialist

Approved by: Carol Ann Carlson
Quality Assurance Manager

Approved by: Shawn A. Keith
Environmental Health and Safety Coordinator

Approved by: Christoph P. Orlandi
Laboratory Director

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held propriety to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any propose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

<i>1. SCOPE AND APPLICATION</i>	2
<i>2. SUMMARY OF METHOD</i>	3
<i>3. DEFINITIONS</i>	3
<i>4. INTERFERENCES</i>	3
<i>5. SAFETY</i>	3
<i>6. EQUIPMENT AND SUPPLIES</i>	4
<i>7. REAGENTS AND STANDARDS</i>	4
<i>8. SAMPLE PRESERVATION AND STORAGE</i>	5
<i>9. QUALITY CONTROL</i>	6
<i>10. CALIBRATION AND STANDARDIZATION</i>	7
<i>11. PROCEDURE</i>	7
<i>12. DATA ANALYSIS AND CALCULATIONS</i>	8
<i>13. METHOD PERFORMANCE</i>	8
<i>14. POLLUTION PREVENTION</i>	9
<i>15. WASTE MANAGEMENT</i>	9
<i>16. REFERENCES</i>	9
<i>17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)</i>	10

1. SCOPE AND APPLICATION

- 1.1. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.
- 1.2. This method is applicable to the acid digestion of soils, sludges, and other solid samples for analysis by Flame Furnace or Inductively Coupled Plasma Spectroscopy (FLAA).

GFAA, or ICP) for all metals except Mercury. It is based on SW846 Method 3050A and the EPA Method referenced in Section 16.

2. SUMMARY OF METHOD

- 2.1. A representative 1 g sample (as received) is digested in HNO₃ and H₂O₂. For GFAA preparation, the digestate is refluxed with HNO₃. For FLAA and ICP preparation, the digestate is refluxed with HCl. The digestate is then diluted to volume. A separate sample is used for dry weight determination.

3. DEFINITIONS

- 3.1. Refer to the glossary in 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 that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

- 5.1. 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. The following specific hazards are known:
 - 5.3.1. The following materials are known to be **corrosive: Nitric Acid, Hydrochloric Acid, Hydrogen Peroxide.**
- 5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**. Solvent and waste containers will be kept closed unless transfers are being made.

- 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. Beakers: 150 mL, 100 mL
- 6.2. Watchglasses
- 6.3. Filter Paper: Qualitative
- 6.4. Calibrated thermometer: 0 - 200°C, used to check hot plate temperature
- 6.5. Hotplate: Capable of maintaining 95°C±4°C
- 6.6. Top Loading Balance: Capable of accurately weighing to 0.01 g
- 6.7. Plastic funnels
- 6.8. Eppendorf repeator and 50 mL tips
- 6.9. Mechanical pipets
- 6.10. Pipet tips: disposable
- 6.11. Tongue blades: wooden
- 6.12. Corning plastic bottles with snap caps
- 6.13. Plastic squirt bottles
- 6.14. Graduated dispenser
- 6.15. Brinkmann bottle top dispenser

7. REAGENTS AND STANDARDS

- 7.1. Reagents
- 7.1.1. Nitric Acid, HNO₃ Concentrated: Reagent grade

7.1.2. Hydrogen Peroxide, H₂O₂, 30%: Reagent grade

7.1.3. Hydrochloric Acid, HCl, Concentrated: Reagent grade

7.1.4. Reagent water

7.2. Standards

7.2.1. Primary Stock Standards

7.2.1.1. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source. Standards must have a purity of 99.5% or greater. Standards may be purchased with individual elements or purchased in mixes.

7.2.1.2. Stock standards solutions must be replaced after one year or sooner if comparison with a second source standard indicates a problem. Standard solutions should be checked frequently for signs of degradation or evaporation.

7.2.2. Laboratory Control Samples (LCS)

7.2.2.1. From the stock standard, prepare a LCS as described in Table I.

7.2.3. Matrix Spike/Matrix Spike Duplicate Standard (MS/MSD)

7.2.3.1. From the stock standard prepare a matrix spike (MS/MSD) as described in Table I.

8. SAMPLE PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers with screw caps at 4°C ± 2°C.

8.3. The holding time is six months from sampling to the completion of analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, MS, MSD, Method Blanks) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank (MB)

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.3. Laboratory Control Sample

9.3.1. One LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. 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.

9.3.2. A midrange LCS must be digested with each batch of samples.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each 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/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. 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/LCSD must be compared to the matrix spike RPD limits.

9.5. Duplicates

9.5.1. A client may request that a sample duplicate and a matrix spike are performed in place of our routine MS/MSD.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation Procedure

11.3.1. Homogenize sample in container with tongue blade.

11.3.2. Weigh and record, to the nearest 0.02 g, 1 g of sample into a beaker. For samples with low percent solids a larger sample size may be used as long as digestion is completed.

11.3.3. Sample Digestion

11.3.3.1. With a squirt bottle, add approximately 5 mL of reagent water and 5 mL concentrated HNO₃, cover with a watchglass, place the beaker on a hotplate at 91-99°C, and reflux 15 minutes **without boiling**. Add 5 mL concentrated HNO₃, replace watchglass and reflux for an additional 30 minutes. Add an additional 5 mL concentrated HNO₃ and reflux for 30 minutes.

CAUTION: Do not allow the sample to go to dryness at any portion of the digestion process. If the sample goes to dryness, the sample must be reprepared.

11.3.3.2. Add approximately 5 - 10 mL of reagent water and 1 mL 30% H₂O₂, **cover with watchglass**, and heat on hotplate. Once effervescing stops, add H₂O₂ in 1 mL increments to a total of 10 mL H₂O₂.

NOTE: Do Not add more than a total of 10 mL 30% H₂O₂

- 11.3.4. For FLAA and ICP preparation, add 5 mL concentrated HCl and reflux for 10 to 15 minutes **without boiling**.
- 11.3.5. For GFAA preparation, continue refluxing for an additional 10 to 15 minutes. Do not add HCl.
- 11.3.6. Remove the beakers from the hot plate and allow them to cool.
- 11.3.7. Filter the sample through quantitative filter paper into a Corning Graduated Plastic Bottle with snap top. Rinse sample's original beaker twice with reagent water. Drain the rinses through the filter. Make a final rinse of the filter with reagent water. Bring the sample final volume to 100 mL with reagent water. Snap top securely on the bottle.
- 11.3.8. The diluted sample has an approximate acid concentration of 5% v/v HCl and 15% v/v HNO₃ (ICP/FLAA/SB), (GFAA 15% v/v HNO₃ - No HCl).

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not Applicable.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. 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 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. Acid waste is disposed in sample prep sink with copious amount of water.
- 15.2. Solid materials (gloves, soiled paper products) are placed in the solid debris container. Do not put liquids in the solid waste container.
- 15.3. Refer to the Laboratory Sample and Waste Disposal plan.
- 15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Quanterra. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

- 16.1. References.
- 16.1.1. SW846, Test Methods for Evaluating Solid Waste. Third Edition, Acid Digestion of Sediments, Sludges, and Soils. Method 3050A
- 16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, Metals, Atomic Absorption Methods, Section 4.1
- 16.2. Associated SOPs
- 16.2.1. Elemental Analysis by Flame Atomic Absorption Spectrophotometry. NC-MT-0001
- 16.2.2. Graphite Furnace Analysis. NC-MT-0002
- 16.2.3. Analysis of Water and Soil Samples by Inductively Coupled Plasma Spectroscopy, NC-MT-0006

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

SPIKING PROTOCOL

Table 1 Spiking Protocol

Preparation Type	Type	Amt of std added LCS & MS/MSD	Initial Sample Vol/Wt	Final Sample Volume
ICP	solid	2 mL ICP-1 5 mL ICP-2	1 ± 0.02g	100 mL
Sb, GFAA	solid	2 mL GFAA	1 ± 0.02g	100 mL
Pb, FLAA	solid	5 mL Pb	1 ± 0.02g	100 mL
Sn, Ti, FLAA	solid	5 mL Sn, Ti	1 ± 0.02g	100 mL
As, Se, Cd, Cr Pb, Tl, Ag, GFAA	solid	2 mL GFAA	1 ± 0.02g	100 mL

STANDARD DEFINITION

Table 2 Standard Definition

Elements	ICP-1	ICP-2	FLAME	GFAA	Ag	Sb
Ag				0.25 ppm	2.5ppm	
Al	100ppm					
As	100ppm			2 ppm		
B	50ppm					
Ba	100ppm					
Be	2.5ppm					
Ca		1000ppm				
Cd	2.5ppm			0.2 ppm		
Co	25ppm					
Cr	10ppm			0.5ppm		
Cu	12.5ppm					
Fe	50ppm					
Hg						
K		1000ppm				
Mg		1000ppm				
Mn	25ppm					
Mo	50ppm					
Na		1000ppm				
Ni	25ppm					

ACID DIGESTION OF SEDIMENTS,
SLUDGES, AND SOILS FOR METAL
ANALYSIS BY GFAA, FLAA, OR ICP

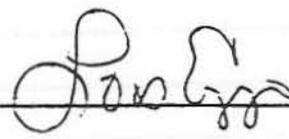
SOP No. NC-IP-0004
Revision No. 1
Revision Date: 02/10/99
Page 14 of 16

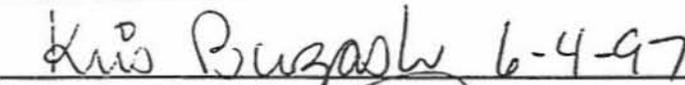
Pb	25ppm		100ppm	2 ppm		
Sb	25ppm			2 ppm		2ppm
Se	100ppm			2 ppm		
Sn	100ppm		100ppm			
Sr	50ppm					
Ti	50ppm		100ppm			
Tl	100ppm			2 ppm		
V	25ppm					
W	50ppm					
Zn	25ppm					

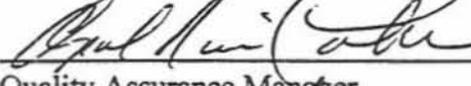
QUANTERRA® STANDARD OPERATING PROCEDURE

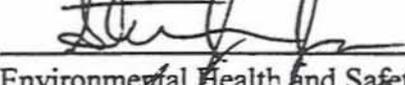
TITLE: SAMPLE RECEIVING

(SUPERSEDES: REVISION 3, 04/18/97)

Prepared by:  6-4-97

Reviewed by:  6-4-97
Technology Specialist

Approved by:  6/4/97
Quality Assurance Manager

Approved by:  6-5-97
Environmental Health and Safety Coordinator

Approved by:  6-4-97
Laboratory Director

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

1. PURPOSE3

2. RESPONSIBILITIES.....3

3. SAFETY.....3

4. PROCEDURES.....4

5. DEFINITIONS10

6. APPENDICES10

1. PURPOSE

- 1.1. The procedures listed in this document describe the responsibilities of Sample Control personnel in ensuring that data is transmitted correctly from the client samples to all personnel involved with sample analysis and review.
- 1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of Sample Receiving and Control personnel to perform the procedures described herein in full compliance with this SOP.
- 2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable personnel to perform this SOP correctly.

3. SAFETY

- 3.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.
- 3.2. 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.
- 3.3. 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.
- 3.4. 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.
 - 3.4.1. The following materials are known to be **corrosive**: **Sulfuric Acid, Nitric Acid, Hydrochloric Acid, Sodium Hydroxide.**

-
- 3.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
 - 3.6. 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.

4. PROCEDURES

- 4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
- 4.2. The Sample Control person will open and examine the shipping container, remove the enclosed sample documents, and record the following information on the Cooler Receipt/ Narrative Form (appendix).
 - 4.2.1. Presence of the custody seals on the outside of the cooler
 - 4.2.2. Presence of the custody papers inside the cooler
 - 4.2.3. The custody papers were properly filled out (ink, signed, match labels)
 - 4.2.4. The custody papers were signed in the appropriate place
 - 4.2.5. Presence of the shipper's packing slip
 - 4.2.6. Presence of packing material information: if yes, type of packing material
 - 4.2.7. Conditions of samples at receipt (chilled, etc.).
 - 4.2.7.1. If temp vial is present, it is used to take the temperature. The temperature of the temp vial is taken as soon as it is removed from the cooler. If a temp vial is not present, proceed to section 4.2.7.2.
 - 4.2.7.2. The temperature of the coolant is recorded if a temp vial is not present. This is performed by placing the thermometer probe between the coolant and the sample(s). If a coolant is not present, proceed to section 4.2.7.3.
 - 4.2.7.3. If no coolant is in the cooler, the thermometer probe is placed between two sample bottles and the temperature recorded.

- 4.2.7.4. If the temperature is outside $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, the anomaly is recorded on the cooler receipt form and the project manager is contacted for further instructions from the client.
- 4.2.8. Bottles sealed in separate plastic bags
- 4.2.9. Condition of bottles upon receipt (good condition, broken, etc.)
- 4.2.10. Complete bottle labels (date, time, client ID)
- 4.2.11. Information on bottle labels and tags agree with custody papers
- 4.2.12. Correct bottles used for the tests indicated
- 4.2.13. VOA bottles were checked for the presence of air bubbles
- 4.2.14. Sufficient amount of sample sent in each bottle
- 4.2.15. Samples were received via overnight courier, client drop off, or other means
- 4.2.16. pH's are taken, on all preserved samples less Volatiles, TOC, and TOX by removing sample lids and using a droplet of sample from in the lid to test the pH. The pH's are then recorded on the cooler receipt form. The pH paper strips are then discarded.
- 4.2.17. If samples are not at the correct pH, the pH is adjusted by adding the appropriate preservative in 5 mL increments up to a maximum of 20 mL or unless there is a reaction. Sulfides are preserved with 6 mL Sodium hydroxide and 2 mL Zinc acetate. The pH adjustment is noted on the cooler receipt form.
- 4.3. The Sample Control person is to remove all sample containers. Any broken, leaking, or dirty sample containers are to be placed inside the fume hood. Dirty sample containers are to be cleaned appropriately, so as not to contaminate the sample storage area. The Sample Control person is to wear disposable latex gloves, safety glasses, and a lab coat while handling any samples.
- 4.4. Any volatile sample(s) suspected (e.g., odor) or known (client information or site history) to be high in volatile concentration, the volatiles department will be contacted for pick-up and segregation of sample(s).
- 4.5. The Sample Control person is to examine all documents and compare information from sample container labels and Chain-of-Custody Records to insure that there is no

discrepancy between documents, ensuring that all documents are properly completed and signed.

- 4.6. If any problems or discrepancies are noted during the sample receiving process that compromise sample integrity, such as limited sample volume, sample identification cannot be determined from the COC, incorrect pH levels (or preservatives if known), or broken, leaking samples, the Project Manager is immediately notified. They in turn will contact the client in an attempt to rectify the situation.
- 4.7. If all samples recorded on the Chain-of-Custody Record were received by the laboratory and there are no problems observed with the sample shipment, the Sample Control person will sign the Chain-of-Custody Record in the "Received for Laboratory by:" box on the document. If problems are noted, sign for shipment and note the problems. All discrepancies are recorded on Cooler Receipt Form.
- 4.8. The Sample Control person will enter each sample into the laboratory computer (QuantIMS), where a unique lot number is assigned to each project received, and sequential sample numbers are designated for each client identification within the lot.
- 4.8.1. Lot Numbers: The lot number is nine characters in length and is based on the date of receipt. Lot number A5J010021 is described as follows:
- A - Quanterra location where the samples were received.
(A = North Canton. B = Tampa. C = Pittsburgh, etc.)
 - 5 - Last digit of the year (i.e. 1995).
 - J - Month (i.e. A = January, B = February, J = October, etc.)
 - 01 - The next 2 numeric characters identify the day of the month, in this case. the first day of the month.
 - 0021 - The next 4 numeric characters are the sequential assignment of numbers specific to each lot received. Each day the first lot logged in receives the number "0001". the second lot receives the number "0002". etc..

For example:

If four bottles were submitted under Client ID numbers AB100-AB103 and the laboratory identification number generated by the computer is A2K100001, then the assigned laboratory number recorded on the Sample Log-In Sheet would be as follows.

<u>Client ID Sample Number</u>	<u>Assigned Laboratory Number</u>
AB 100	A2K100001-001
AB 101	A2K100001-002
AB 102	A2K100001-003
AB 103	A2K100001-004

- 4.8.2. **Sample Numbers:** The samples in each lot are assigned a sample number that is attached to the lot number and are reset at each new lot. For example: the first and second samples in the lot above are labeled A5J010121-001 and A5J010121-002.
- 4.8.3. **Sample Suffixes:** Each sample also has a 1 character field (which is not a required field for all samples) called the suffix which identifies the sample as specified below.

Client Sample	no suffix
Method Blank	B
Laboratory Control Sample	C
Laboratory Control Sample Duplicate	L
Matrix Spike	S
Matrix Spike Duplicate	D
Sample Duplicate	X
Serial Dilution	P
Sample Confirmation	Y

Post Digestion Spike	Z
Re-analysis	I

Example: A5J010121-001X is a sample duplicate for sample A5J010121-001.

- 4.8.4. Work Order Numbers: Each test requested by the client for an individual sample receives an individual 8 digit work order number assigned by QuantIMS. Work order number A5WE1-2-1C is described as follows:

A5WE1 - In addition to the three digit sample identification described in 4.7.2 (i.e. - 001 and - 002), the first 5 characters of the work order number also identifies each unique sample. This identification is generated in QuantIMS using a sequential logic that is beyond the scope of this SOP to describe.

2 - The "modifier" indicates the type of run. In this case this is the second time the sample had to be run. If it needs reprep and run again, the number would indicate a "3". The original analysis work order number assigns "1" to the modifier position.

1C - The "suffix" is the identification of the specific test for that sample. The suffix in this case is not always sequential, but is unique to the test to be performed on the sample.

Example: A5WE1-2-1C is the assigned 8 digit work order number for the reanalysis of the chloride test on the sample A5WE1. A5WE1-1-05 could be the 8 digit work order number for the analysis of SW846 8270 on sample A5WE1.

- 4.9. Once all sample containers have been properly labeled and all the information has been recorded by the Sample Lot Summary, the Sample Control person will place the samples into the proper storage locations. These locations are as follows:
- 4.9.1. Organic extractable samples (Semivolatiles, Pesticides/PCBs) are to be placed into the walk-in refrigerators located in Sample Receiving.
- 4.9.2. Volatile samples are to be stored in the two double-door refrigerators located in the Sample Custodian area. One refrigerator is for MS Volatile samples and the second is for GC Volatiles samples.
- 4.9.2.1. Samples known or suspected to be of high concentration are not stored in these refrigerators. The applicable volatile group is contacted to obtain the samples for segregation.

4.9.3. Inorganic samples are to be placed into the walk-in refrigerators located in Sample Receiving.

4.9.4. Metals samples are placed in a non-refrigerated room located in the Sample Custodian area.

5. SAMPLE CONTROL RECORD

5.1. For clients who request a show of sample transfer from sample receipt to storage, a sample control record is completed (see figure 8.2.4). This record is also referred to as an internal chain of custody (COC). The form is completed as follows:

5.1.1. Laboratory sample number - record/list the individual five digit work order number in this column. List all pertaining samples in the project lot. Use an additional sheet if necessary. Do not record multiple lots on a sample control record.

5.1.2. Transferred by - record name of person making the transfer

5.1.3. Date - record date of sample transfer

5.1.4. Entered - "X" or "√" since samples are already logged into the LIMs.

5.1.5. Removed - leave blank since samples are not being removed

5.1.6. Reason - record "storage"

5.1.7. Date returned - leave blank

5.2. The completed sample control record is attached to the summary package.

6. SUBCONTRACTING OF SAMPLES

6.1. Samples that are logged but not analyzed at the laboratory are subcontracted to different laboratories for analysis including other Quanterra facilities.

6.2. The LIMs system will automatically print a Sample Analysis Requisition for these samples upon completion of the log-in process (see figure 8.2.5).

6.3. This form contains information necessary for sample analysis. The original form is sent to the subcontracted laboratory and a copy is attached to the summary package. The

Sample Analysis Requisition form must have a relinquished signature with a date and time. Any additional information necessary for sample analysis must be handwritten on the form (e.g. list of compounds, homogenizing of samples, limited quantity, etc.). In order to track subcontracted samples, the lab purchase order number on the Sample Analysis Requisition form must be recorded in the subcontracted sample PO book located in the receiving log-in area.

7. DEFINITIONS

7.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP)

8. APPENDICES

8.1. Comments

8.1.1. The only personnel authorized to execute this SOP are the Sample Log-In persons.

8.1.2. Wherever "Sample Control" is mentioned in all SOPs, it is assumed to include the sample custodian or any alternate that is designated by the Sample Control Coordinator.

8.2. Appendix:

8.2.1. Cooler Receipt Form/Narrative

8.2.2. Preservative Preparation

8.2.3. Preservative Requirements

8.2.4. Internal Chain of Custody

8.2.5. Sample Analysis Requisition

8.2.6. Subcontracted PO Logbook

Cooler Receipt/Narrative Form

Quanterra® Cooler Receipt Form/Narrative

North Canton Facility

Client: _____ Project: _____ Quote#: _____
Cooler Received on: _____ Opened on: _____ by: _____
Fedx Client Drop Off UPS Airborne Other: _____ (Signature)
Cooler Safe Foam Box Client Cooler Other: _____
Quanterra Shipper No#: _____

1. Were custody seals on the outside of the cooler and intact? Yes No
If YES. Quantity _____ Location _____
Were signature and date correct? Yes No NA
2. Shipper's packing slip attached to this form? Yes No
3. Were custody papers included inside the cooler and relinquished? Yes No
4. Did you sign the custody papers in the appropriate place? Yes No
5. Packing material used:
Peanuts Bubble Wrap Vermiculite Foam None Other: _____
6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temp)
METHOD: Temperature Vial Coolant Against Bottles
COOLANT: Wet Ice Blue Ice Dry Ice None
7. Were all the bottles sealed in separate plastic bags? Yes No
8. Did all bottles arrive in good condition (Unbroken)? Yes No
9. Did all bottle labels and tags agree with the custody papers? Yes No
10. Were samples at the correct pH? Yes No NA
11. Were correct bottles used for the tests indicated? Yes No
12. Were air bubbles present in any VOA vials? Yes No NA

SAMPLE RECEIVING

SOP No. NC-SC-0005
 Revision No. 4
 Revision Date: 06/02/97
 Page 13 of 26

13. Was a sufficient amount of sample sent in each bottle?		Yes <input type="checkbox"/> No <input type="checkbox"/>
Contacted PM _____ Date: _____ by: _____ via Voice Mail <input type="checkbox"/> Verbal <input type="checkbox"/> Other <input type="checkbox"/>		
Concerning: _____		
Check <input checked="" type="checkbox"/>	MACRO	MACRO
1. CHAIN OF CUSTODY		
	SR1A	Samples were received under proper custody procedures and without discrepancies.
	SR1B	The chain of custody and sample bottles did not agree. The following discrepancies occurred _____
2. SAMPLE CONDITION		
	SR2A	Sample(s) _____ were received or requested after the recommended holding time had expired.
	SR2B	Sample(s) _____ were received with insufficient volume
	SR2C	Sample(s) _____ were received in a broken container.
	SR2D	Sample(s) _____ were received in unapproved containers.
3. SAMPLE PRESERVATION		
<input checked="" type="checkbox"/>	SR3A	The temperature of the cooler was _____ °C
	SR3B	Sample(s) _____ received for Volatile analysis was/were received with headspace.
	SR3C	Sample(s) _____ were received incorrectly preserved and were adjusted accordingly in sample receiving.
	SR3D	Sample(s) _____ were received incorrectly preserved and split off in sample receiving.
	SR3E	Samples (s) _____ were received incorrectly preserved and were unable to be analyzed.
	N A	See back for other anomalies

Preservative Preparation

NOTE

Preservative Preparation

- 4N Sodium Hydroxide Add 12.0 g NaOH pellets to 800 mL reagent water in a 1000 mL volumetric flask. Mix, cool, and dilute to volume with reagent water. Store in a well labeled plastic bottle.
- 1N Zinc Acetate Add 55 g $Zn(C_2H_3O_2) \cdot H_2O$ to 200 mL reagent water in a 250 mL volumetric flask. Mix and dilute to volume with reagent water. Store in a well labeled plastic bottle.
- 1:1 Hydrochloric Acid (18%): Slowly add 1000 mL concentrated HCl to 1000 mL reagent water and mix. Store in a well labeled plastic coated acid bottle.
- 1:4 Nitric Acid (18%): Slowly add 360 mL concentrated HNO_3 to 1640 mL reagent water and mix. Store in a well labeled plastic coated acid bottle.
- 1:2 Sulfuric Acid (33%): In a 2000 mL beaker, SLOWLY and CAREFULLY add 500 mL concentrated H_2SO_4 to 1000 mL reagent water and mix. A cool water bath may be needed to cool the solution and beaker. Store in a well labeled plastic acid bottle.

NOTE:

All preparations must be performed in a hood and proper personal protective equipment must be worn. All reagents and final preservative solution must be documented in applicable reagent logbooks.

Preservatives, Containers, and Volumes

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative ^{1,2}	Volume	Parameter	Container	Preservative ^{1,2}	Volume
Asbestos	P	None	250 mL	Radiological	P	HNO ₃	4 L
				Alpha, Beta, Radium			
Acidity	P	None	250 mL	Hardness	P	HNO ₃	250 mL
Alkalinity (Sep)	P	None	250 mL	Metals	P	HNO ₃	1 L
BOD	P	None	250 mL	Dissolved Metals*	P	HNO ₃	1 L
Carbonaceous BOD	P	None	250 mL	Total Organic Carbon (TOC)	G	HCl	2 x40 mL
Bromide (Br)	P	None	250 mL	Chemical Oxygen Demand	P	H ₂ SO ₄	250 mL
Chloride (Cl)	P	None	250 mL	Total Organic Halogens	G	H ₂ SO ₄	250 mL
Chromium. 6 ⁺	P	None	250 mL	COD	P	H ₂ SO ₄	250 mL
R. Chlorine	P	None	100 mL	Ammonia Nitrogen (NH ₃)	P	H ₂ SO ₄	500 mL
Color	P	None	50 mL	TKN	P	H ₂ SO ₄	1L
Conductivity	P	None	250 mL	Nitrate/Nitrite	P	H ₂ SO ₄	250 mL
Corrosivity	P	None	250 mL	Oil & Grease	G	H ₂ SO ₄	1 L
Dissolved Oxygen	G	None	300 mL	Phenols	G	H ₂ SO ₄	1 L
Fecal Coliform	P	None	125 mL	Total Phosphorus	P	H ₂ SO ₄	250 mL
Flashpoint	G	None	100 mL	TON	P	H ₂ SO ₄	1 L
Fluoride	P	None	250 mL				
Nitrate	P	None	250 mL	TRPH - IR 418.1	G	HCl	2 L
Nitrite	P	None	250 mL	VOC 601	G	HCl	3x40 mL
pH	P	None	50 mL	VOC 8010	G	HCl	3x40 ml
Elemental PO ₄	G	None	250 mL	VOC 624	G	HCl	8x40 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative ^{1,2}	Volume	Parameter	Container	Preservative ^{1,2}	Volume
Orthophosphate	P	None	250 mL	BTEX 8020	G	HCl	3x40 mL
TDS	P	None	250 mL	VOC 8240	G	HCl	3x40 mL
TSS	P	None	250 mL	THM/502.2	G	HCl	2x40 mL
Total Solids	P	None	250 mL	502.2	G	HCl & Asc. Acid	2x40 mL
TVS	P	None	250 mL	VOC 624	G	HCl	3x40 mL
T. Coliform	P	None	125 mL	VOC 602	G	HCl	3x40 mL
Settleable Solids	P	None	1L	465 C & D	G	HCl	4x40 mL
Silica	P	None	250 mL	BTEX 8021	G	HCl	3x40 mL
Sulfate	P	None	250 mL	VOC	G	HCl	3x40 mL
Sulfite	P	None	250 mL	VOC 8260	G	HCl	3x40 mL
Surfactants (MBAS)	P	None	250 mL	VOC and VOA	G	HCl	3x40 mL
Turbidity	P	None	250 mL	VOC 8010/8020	G	HCl	3x40 mL
TPH-GC	G	None	2 L				
				Total Cyanide	P	NaOH ³	250 mL
BNAs	G	None	2 L	Amenable Cyanide	P	NaOH	250 mL
BNA + Dioxin	G	None	2 L	Free Cyanide	P	NaOH	250 mL
PN/PAH	G	None	2 L	Sulfide	P	Zn Acetate & NaOH	1 L
Pesticides	G	None	2 L	Formaldehyde	G	None	500 mL
Reactive Cyanide	P	None	1 L	Carbonate	P	None	250 mL
Reactive Sulfide	P	None	1 L	Bicarbonate	P	None	250 mL
PCB	G	None	2 L	TPH - Diesel (EXT)	G	None	2 L

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative ^{1,2}	Volume	Parameter	Container	Preservative ^{1,2}	Volume
Pesticides + PCBs	G	None	2 L	TPH - Gasoline (P&T)	G	HCl	2x40 mL
Herbicides	G	None	2 L	Glycols 8015	G	None	2x40 mL
OPPs	G	None	2 L	BTEX & MTBE	G	HCl	3x40 mL
				601/602	G	HCl	3x40 mL

* Filtered in field

¹ HCl, HNO₃, and H₂SO₄ to pH < 2. NaOH to pH > 12

² Temperature = 4°C ± 2°C except for aqueous metals

³ Samples to be analyzed for Cyanide should be field-filtered for Residual Chlorine. If Residual Chlorine is detected, ascorbic acid (0.6 g) should be added.

Internal Chain of Custody

SAMPLE RECEIVING

SOP No. NC-SC-0005
Revision No. 4
Revision Date: 06/02/97
Page 22 of 26

Sample Analysis Requisition

SAMPLE RECEIVING

SOP No. NC-SC-0005
Revision No. 4
Revision Date: 06/02/97
Page 24 of 26

Example Subcontracted PO Logbook

SAMPLE RECEIVING

SOP No. NC-SC-0005

Revision No. 4

Revision Date: 06/02/97

Page 26 of 26

Controlled Copy
Copy No: _____
Implementation Date _____

SOP No: CORP-GC-0001
Revision No: 5
Revision Date: 12/7/98
Page 1 of 22

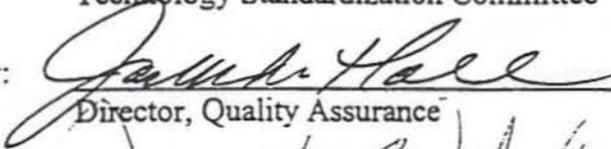
QUANTERRA® STANDARD OPERATING PROCEDURE

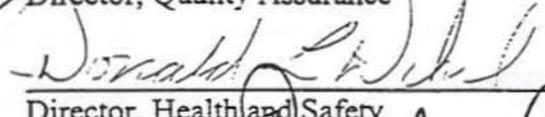
TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B,
8021B, 8081A, 8082 and 8151A, SW-846

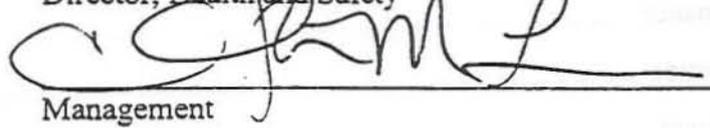
(SUPERSEDES: Revision 4)

Prepared by: Richard Burrows

Reviewed by: 
Technology Standardization Committee

Approved by: 
Director, Quality Assurance

Approved by: 
Director, Health and Safety

Approved by: 
Management

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra® Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's® qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra®.

The user agrees by its acceptance or use of this document to return it upon Quanterra's® request and not to reproduce, copy, lend or otherwise dispose or disclose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

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	5
7.	Reagents and Standards	5
8.	Sample Preservation and Storage.....	6
9.	Quality Control	6
10.	Calibration and Standardization.....	11
11.	Procedure	16
12.	Data Analysis and Calculations	19
13.	Method Performance.....	23
14.	Pollution Prevention	24
15.	Waste Management.....	24
16.	References.....	24
17.	Miscellaneous	24

List of Appendices:

Appendix A	Analysis of Volatile Organics based on Methods 8010B, 8020A, and 8021A
Appendix B	Analysis of Organochlorine Pesticides and PCBs based on Method 8080A
Appendix C	Analysis of Phenoxy Acid Herbicides based on Method 8150B
Appendix D	Analysis of Organochlorine Pesticides and PCBs based on Method 8081

List of Tables

Table A1	Standard analyte list for 8010B, 8020A and 8021A
Table A2	Recommended conditions for method 8020A
Table A3	Recommended conditions for method 8010B
Table A4	Recommended conditions for method 8021A
Table A5	Surrogate and Internal standard concentrations for aqueous and low level soil samples, methods 8010B, 8020A, and 8021A
Table A6	Surrogate and internal standard concentrations for medium level soil samples, methods 8010B, 8020A and 8021A
Table A7	Concentrations for LCS and MS/MSD compounds, low level soil and aqueous, methods 8010B, 8020A and 8021A.
Table A8	Concentrations for LCS and MS/MSD compounds, medium level soil, methods 8010B, 8020A and 8021A
Table A9	Continuing calibration and initial demonstration limits, method 8010B
Table A10	Continuing calibration and initial demonstration limits, method 8020A
Table B1	Standard analyte and reporting limits, method 8080A
Table B2	Recommended conditions, method 8080A
Table B3	Calibration levels, method 8080A
Table B4	Column degradation evaluation mix, method 8080A
Table B5	LCS/matrix spike and surrogate levels, method 8080A
Table B6	LCS/matrix spike and surrogate levels for Aroclor analysis, method 8080A
Table B7	LCS/matrix spike and surrogate levels for TCLP, method 8080A
Table B8	Suggested analytical sequence, method 8080A
Table B9	Performance limits, method 8080A
Table C1	Standard analyte list, method 8150B
Table C2	Instrumental conditions, method 8150B
Table C3	Calibration standards, method 8150B
Table C4	LCS/Matrix spike and surrogate levels, method 8150B
Table C5	Performance limits, method 8150B

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 are prepared for analysis using continuous or separatory funnel liquid / liquid extraction (SOP # CORP-OP-0001) or sonication or soxhlet 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 appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned

immediately. Refer to the Quanterra Chemical Hygiene plan for a complete description of personal protection equipment.

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.

5.1.1. Opened containers of neat standards will be handled in a fume 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 gases are stored at -10 to -20°C. Other volatile and semivolatile stock standard solutions are stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. 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. (6 months if also used for 600 series methods). Stock standards of gases must be replaced at least every 2 months. (Every week if also used for 600 series methods). Other volatile stock standards must be replaced at least every 6 months (1 month if used for 600 series analysis) or sooner if comparison with check standards prepared from an independent source indicates a problem.

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.

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ 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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and analyzed within 40 days of the start 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. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate.

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 annually. The recovery limits are mean recovery ± 3 standard deviations for surrogates and LCS, and mean recovery ± 2 standard deviations for matrix spikes. Precision limits for matrix spikes / matrix spike duplicates are zero to mean relative percent difference + 2 standard deviations.

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.

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 rules for which column's result to report are the same as for samples (Section 12.1). That is, the lower of the two results 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 reparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reparation 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 9.5 mL of methanol as described in section 11.5 of 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 done 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 analysed with any sequence that does not contain a method blank.

9.6.2. A new sequence starts with any new initial calibration and if there has been a break of greater than 12 hours in sample analysis.

9.6.3. An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.6.4. 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. 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 reparation 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 rules for which column's result to report are the same as for samples (Section 12.1). That is, the lower of the two results 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 that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.8.1. If dual column analysis is used, the rules for which column's result to report are the same as for samples (Section 12.1). That is, the lower of the two results 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. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in 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 and any changes in instrument operating parameters, including gas flows, detector temperatures, oven temperatures, etc.
- 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.
- 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.
- 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.

Equation 1

$$\text{Calibration Factor (CF)} = \frac{\text{Area or Height of Peak}}{\text{Mass Injected (ng)}}$$

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. Use of peak area or height must be consistent. It is not permitted to switch between using peak area and height for quantitation within an analytical sequence.

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 there is interference, the external standard approach must be used. 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:

Equation 2

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

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. Linear regression is a special case of the quadratic fit and may be used if the calibration range is sufficiently narrow so that a straight line will fit the calibration points. Average response factor may be used if the % RSD of the response factors or calibration factors is < 20%.

10.6.1. Average response factor

The average response factor may be used if the percent relative standard deviation (%RSD) of the response factors is < 20%.

The equation for average response factor is:

Equation 3

$$\text{Average response factor} = \frac{\sum RF_{1-n}}{n}$$

Where: n = Number of calibration levels

$\sum RF_{1-n}$ = Sum of response factors for each calibration level

10.6.2. Linear regression

The linear fit uses the following functions:

10.6.2.1. External Standard

Equation 4

$$\text{Concentration} = A + BR$$

Where: A = Intercept
B = Slope
R = Response

10.6.2.2. Internal Standard

Equation 5

$$\text{Concentration} = A + B \frac{(R \times C_s)}{R_s}$$

Where the variables are defined in equations 2 and 4

10.6.3. Quadratic curve

The quadratic curve uses the following functions:

10.6.3.1. External standard

Equation 6

$$\text{Concentration} = A + BR + CR^2$$

10.6.3.2. Internal Standard

Equation 7

$$\text{Concentration} = A + B\left(\frac{R \times C_{is}}{R_{is}}\right) + C\left(\frac{R \times C_{is}}{R_{is}}\right)^2$$

Where: C = Curvature

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard deviation (%RSD) 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 deviation is calculated as follows:

Equation 8

$$\% \text{ RSD} = 100\% \times \sqrt{\frac{\sum_{i=1}^N \left[\frac{C_i - PC_i}{C_i} \right]^2}{(N - P)}}$$

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, this equation gives the same value as the %RSD of the response factors.

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 the reporting limit for the analyte.
- Relative standard deviation of the calibration points from the curve used must be < 20%.

- Some data systems will not measure the %RSD from a linear or quadratic fit. In this case, the correlation coefficient may be used as an alternative to the %RSD, and must be greater than 0.995.

10.8.1. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.9. Calibration Verification

10.9.1. Continuing Calibration

The working calibration curve or RF must be verified by the analysis of a mid point continuing calibration standard at the beginning, after every 10 samples, and at the end of the analysis sequence (QC and instrument blanks included).

10.9.2. Daily Calibration

At least every 24 hours a daily calibration must be analyzed. The requirements of the daily calibration are the same as the continuing calibration with the addition that retention times are updated.

10.9.3. Any analyte that is reportable as found must have a % difference of $\leq 15\%$ in the preceding continuing calibration, on the column used for quantitation. For dual column analysis, the column used for quantitation will be the column with the lower result. Methods 8010B and 8020.A have different continuing calibration limits that are obtained from Table 3 of the reference method and are listed in Appendix A of this SOP.

10.9.4. For any analyte that is not reportable as found, the % difference may be -15% to -30% .

10.9.5. Reportable as found is defined as any analyte that would be reported as anything other than a non-detect.

-
- 10.9.6. If dual column analysis is used, at least one column must meet the criteria listed above. The other column must be within $\pm 30\%$ difference from the initial calibration.
 - 10.9.7. It is not necessary to run a continuing calibration standard at the beginning of the sequence if the first 10 samples are analyzed immediately after the completion of the initial calibration.
 - 10.9.8. The last sample in the sequence must be followed by an ending calibration. The ending calibration serves the analyst in judging the validity of the sequence.
 - 10.9.9. Methods 8010B and 8020A have different continuing calibration criteria. Criteria for continuing calibrations for these methods can be found in the appendices.
 - 10.9.10. % Difference calculation

Equation 9

$$\% \text{ Difference} = \frac{\text{Calculated value} - \text{Expected value}}{\text{Expected value}} \times 100\%$$

10.9.11. Corrective Actions for Continuing Calibration

If the % difference for any analyte is $> +30$ to -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 response for any analyte still varies by more than 30%, a new calibration curve must be prepared.

10.9.12. Corrective Action for Samples

Any samples injected *after* the standard exceeding the continuing calibration criteria 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, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with 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 one major peak. 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 daily 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

11.6.5.1. The retention times of all compounds in each continuing calibration must be within the retention time windows established by the daily calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed 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 daily calibration standards. (See the method 8080A appendix for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each daily calibration, but not for the continuing calibration 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 flags required). For confirmed results, the lower of the two results is reported.

12.1.2. If the % difference between the response on the two columns is greater than 50%, 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.

12.1.3. Multi-response Analytes (Aroclors)

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.

12.1.4. 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 the baseline rise is less than half the height of the peaks in the level 3 standard, then the sample should be reanalyzed at a more concentrated dilution.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Samples

If internal standard calibration is used, then the internal standard response must be within 50 to 200% of the response in the preceding continuing calibration standard.

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.

12.7. External Standard Calculations

12.7.1. Aqueous Samples

Equation 10

$$\text{Concentration } (\mu\text{g} / \text{L}) = \frac{(A_s \times V_i \times D_i)}{(CF \times V_t \times V_s)}$$

Where:

A_s = Response for the analyte in the sample

V_i = Volume of extract injected. μL .

D_i = 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

12.7.2. Non-aqueous Samples

Equation 11

$$\text{Concentration } (\mu\text{g} / \text{kg}) = \frac{(A_x \times V_i \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \% \text{Moisture}}{100} \quad (D = 1 \text{ if wet weight is required})$$

A_x , V_i , D_f , CF and V_i are as defined in Equation 10

12.8. Internal Standard Calculations

12.8.1. Aqueous Samples

Equation 12

$$\text{Concentration } (\mu\text{g} / \text{L}) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng

A_{is} = Response of the internal standard

RF = Response factor for analyte

A_x , D_f , V_s are as defined in Equation 10

12.8.2. Non-aqueous Samples

Equation 13

$$\text{Concentration } (\mu\text{g} / \text{kg}) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

All variables are as defined in equations 11 and 12

12.9. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

Equation 14

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

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 facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update 1, July 1992. Section 8000A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

- 17.1.1. Section 7.6.8 of Method 8000A in SW-846 recommends reanalysis if the continuing calibration does not meet criteria and "if the initial analysis indicated the presence of specific target analytes that exceeded the criterion." This SOP is more rigorous than the requirements in SW-846 in that reanalysis is required if the continuing calibration exceeds - 15 to + 30% difference, whether or not the initial analysis indicated the presence of specific analytes.
- 17.1.2. Method 8000A in SW-846 recommends that the continuing calibration be within 15% difference from the calibration curve for all analytes. This SOP allows for a % Difference of + 30% and -15% for analytes that are not detected. This is supported by the statement in Method 8000A that reanalysis is only required "if the initial analysis indicated the presence of specific target analytes that exceeded the criterion." In any event, the % difference of the continuing calibration must be $\leq 15\%$ for any analyte that is to be quantitated and reported.
- 17.1.3. Chapter I 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.

- 17.1.4. Retention time windows are calculated as in SW-846. However, if a retention time window of less than 0.05 min. is calculated, the window defaults to 0.05 min.
- 17.1.5. Retention time windows are updated each day using the daily standard, as required by method 8000A. The system is not recalibrated unless the continuing calibration standards fall outside the retention time windows set by the daily standard.

17.2. Modifications from Previous Revision

- 17.2.1. Directions for methods 8081 and 8151 have been added.

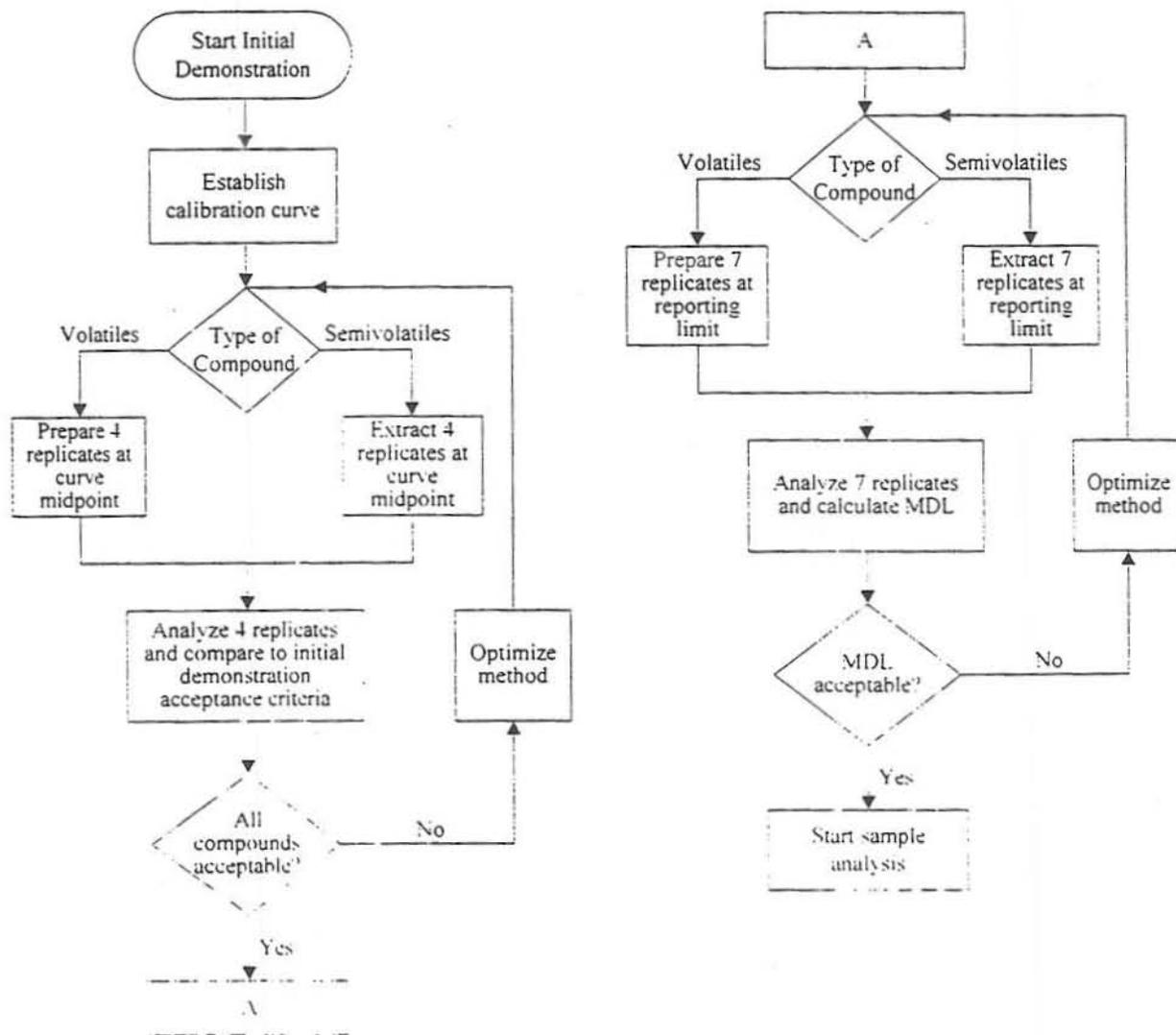
17.3. Facility Specific SOPs

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.

- 17.3.1. *Refer to the SOP change form on file in North Canton's Quality Assurance office.*

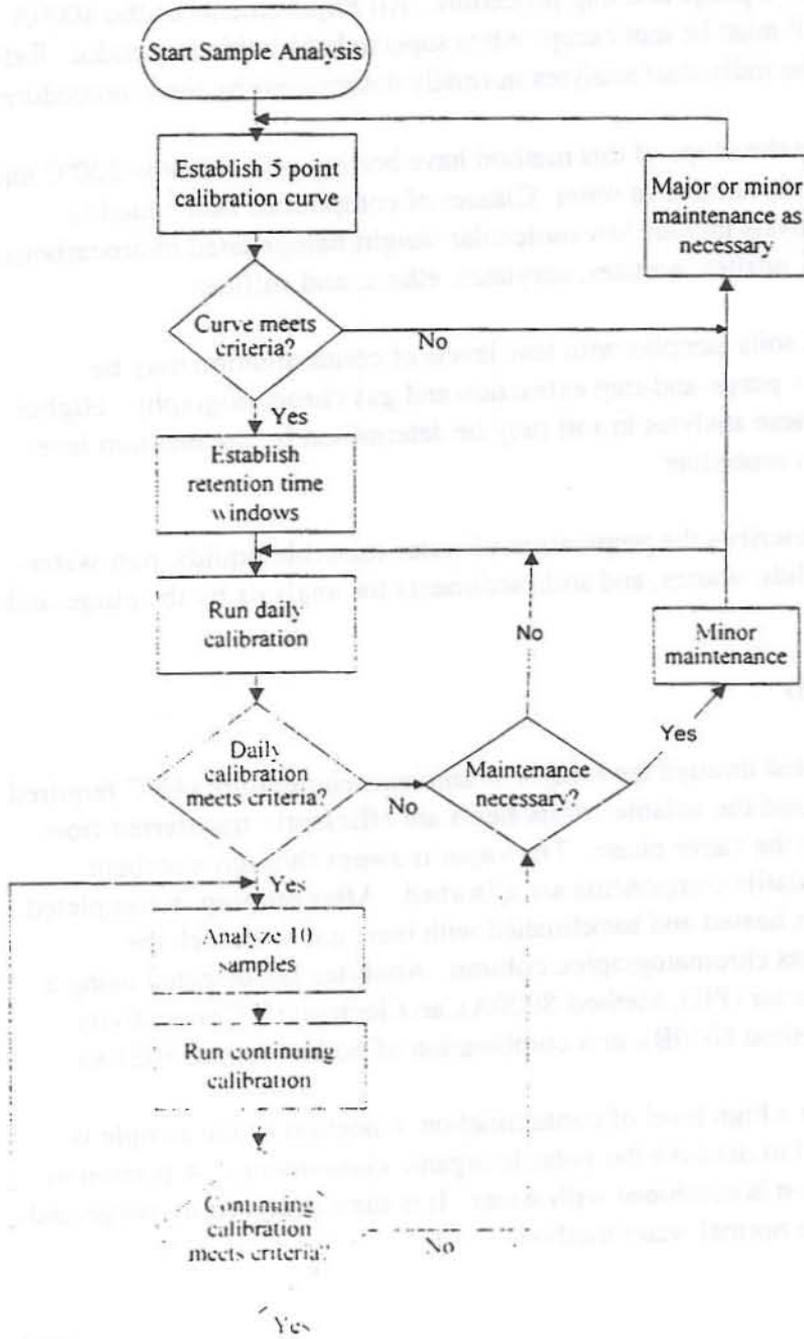
17.4. Flow Diagrams

17.4.1. Initial demonstration and MDL¹



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. Sample Analysis¹



¹ This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

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. All requirements of the 8000A 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 (40°C required for low level soils), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a 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 (PID, Method 8020A), an Electrolytic Conductivity Detector (ELCD, Method 8010B), or a combination of both (Method 8021A).
- 2.2. For soil samples with a high level of contamination, 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.

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 8000A 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 8000A 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

6. EQUIPMENT AND SUPPLIES

- 6.1. Microsyringes -- 10uL, 25uL, 100uL, 250uL, 500uL, and 1000uL. 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

**ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A**

6.10. Balance capable of weighing to 0.01g for samples.

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000A 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. Methanol -- Purge and Trap Grade

7.4. Standards

Refer to tables A-5, A-6, A-7 and A-8 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.

7.4.1. 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. 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.

8.2. Solid samples are not preserved.

8.3. All samples are stored in glass containers with Teflon lined septa at 4°C - - 2°C, with minimum headspace.

**ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A**

- 8.4. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.
- 8.5. 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. The lack of preservation should be addressed in the case narrative.
- 8.6. 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.

9. QUALITY CONTROL

- 9.1. Refer to the method 8000A 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 8000A section of this SOP, section 10, for general calibration procedures. The %RSD limits for the initial calibration are given in Tables A-9 and A-10.

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 method 8021A, a single column is used and the PID and ELCD detectors are connected in series. This configuration may also be used for simultaneous 8010B/8020A determination.

- 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 8000A section of this SOP for details of initial calibration criteria.

**ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A**

- 10.3.2. Soil samples must be purged at 40°C, therefore the calibration curve must also be purged at 40°C. A separate calibration, purged at ambient temperature, must be used for aqueous samples. The aqueous calibration may be used for medium level soils.
- 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. Continuing Calibration

- 10.4.1. Refer to Tables A-9 and A-10 for details of continuing calibration acceptance criteria.
- 10.4.2. The level 3 calibration standard is used for the continuing calibration.

11. PROCEDURE

- 11.1. Refer to the method 8000A 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 daily calibration that meets % difference criteria from an existing initial calibration.

- 11.2.1. The daily calibration must be analyzed at least once every 24 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 24 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.
- 11.2.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.
- 11.2.3. After every 10 samples (including LCS, method blank, and MS samples) a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest for the volatile methods.

It is not necessary but it is permissible to update retention time windows with continuing calibrations.

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. For method 8021A confirmatory information 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. Spike with the appropriate volume of surrogate/internal standard solution and matrix spike solution (if required) through the barrel of the syringe. Refer to Tables A-5, A-6, A-7 and A-8 for volumes and concentrations of spiking solutions.
- 11.4.5. Load onto the purge and trap device and start the run.
- 11.4.6. 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.

**ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A**

11.4.7. Dilutions may be made in gas tight syringes unless the volume of sample used is less than 5 μ L, in which case dilution in volumetric flasks will be necessary.

11.4.7.1. Spike with the same volume of surrogate/internal standard solution as used for undiluted samples prior to loading onto the purge and trap device.

11.4.7.2. For Matrix spike / matrix spike duplicates where the sample requires dilution, the sample is spiked after the dilution is performed.

11.5. Low-Level Solids Analysis

This method is based on purging a heated sediment/soil sample mixed with organic free 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.

11.5.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.5.2. 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 0.5 g. If the sample is contaminated with analytes such that a purge amount less than 0.5 g is appropriate, use the medium level method described in section 11.6.

11.5.3. Connect the purge vessel to the purge and trap device.

11.5.4. 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.

11.5.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.5.6. Add the heater jacket or other heating device and start the purge and trap unit.

**ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A**

- 11.5.7. Soil samples that have low IS recovery when analyzed should be reanalyzed once to confirm matrix effect.
- 11.6. Medium-level Soil Analysis: This method is based on extracting the soils with methanol. A waste sample is either extracted or diluted, depending on the solubility with methanol. An aliquot of the extract is added to organic free water containing internal standards as appropriate. These extracts are analyzed under the same conditions as aqueous samples.
- 11.6.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.
- 11.6.2. Weigh 4 g (wet weight) of sample into a tared 20 mL vial. Note and record the actual weight to 0.1 g.
- 11.6.3. Quickly add 9.5 mL of methanol and 0.5 mL of surrogate solution. For matrix spikes or LCS, add 9.0 mL of methanol, 0.5 mL of surrogate solution, and 0.5 mL of matrix spiking solution. For method blanks, use 9.5 mL of methanol spiked with 0.5 mL of surrogate solution. Cap the vial.
- 11.6.4. Shake the vial for 2 min.
- 11.6.5. Pipet approximately 1.0 mL of the extract into a 1 or 2 mL screwtop autosampler vial for storage, using a disposable pipet. The remainder may be discarded.
- 11.6.6. 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 μ L for a 5 mL purge) methanolic extract to the syringe. Add internal standard if used. Load the sample onto the purge and trap device. If less than 5 μ L of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 μ L will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

Refer to section 12 of the 8000A 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 8000A section of this SOP are presented in Tables A9 and A-10. The spiking level should be 20 μ g/L.

**ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A****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 according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods, SW 846, 3rd Edition, Final Update II, September 1994. Sections 8010B, 8020A and 8021A.

17. MISCELLANEOUS**17.1. Modifications from Reference Method**

17.1.1. Purge volumes greater than 5 mL (up to 25 mL) may be used as required to meet reporting limits.

17.1.2. The shutoff valve for the sample-measuring syringe has been omitted.

17.1.3. The use of a soil aliquot between 1 g and 5 g is allowed if performance criteria are met.

17.1.4. The method has been extended to include the use of VOA vial sampling purge and trap autosamplers.

17.2. Modifications from previous revision

17.2.1. Recommended internal and surrogate standards have been changed.

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.

17.3.1. Refer to the SOP change form on file in North Canton's Quality Assurance office.

17.4. Tables

Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
8010B	Bromodichloromethane	75-27-4	1.0	1.0	120
	Bromoform	75-25-2	1.0	1.0	120
	Bromomethane	74-83-9	1.0	1.0	120
	Carbon Tetrachloride	56-23-5	1.0	1.0	120
	Chlorobenzene	108-90-7	1.0	1.0	120
	Chloroethane	70-00-3	1.0	1.0	120
	2-Chloroethyl vinyl ether	110-75-8	5.0	5.0	620
	Chloroform	67-66-3	1.0	1.0	120
	Chloromethane	74-87-3	1.0	1.0	120
	Dibromochloromethane	124-48-1	1.0	1.0	120
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	120
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	120
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	120
	Dichlorodifluoromethane	75-71-8	1.0	1.0	120
	1,1-Dichloroethane	75-34-3	1.0	1.0	120
	1,2-Dichloroethane	107-06-2	1.0	1.0	120
	1,1-Dichloroethene	75-45-4	1.0	1.0	120
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	120
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	120
	Dichloromethane(DCM)	75-09-2	5.0	5.0	620
	1,2-Dichloropropane	78-87-5	1.0	1.0	120
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	120
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	120
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	120
	Tetrachloroethene	127-18-4	1.0	1.0	120
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	120
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	120
	Trichloroethene	79-01-6	1.0	1.0	120
	Trichlorofluoromethane	75-69-4	1.0	1.0	120
	Vinyl Chloride	75-01-4	1.0	1.0	120
Additional	Benzyl Chloride	100-44-7	5.0	5.0	620

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Table A-1					
Standard Analyte List					
Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
analytes for 8010B	Bromobenzene	108-86-1	1.0	1.0	120
	Dibromomethane	74-95-3	1.0	1.0	120
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	120
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	120
8020A	Benzene	71-43-2	1.0	1.0	120
	Chlorobenzene	108-90-7	1.0	1.0	120
	1,2-Dichlorobenzene	75-34-3	1.0	1.0	120
	1,3-Dichlorobenzene	107-06-2	1.0	1.0	120
	1,4-Dichlorobenzene	75-45-4	1.0	1.0	120
	Ethyl Benzene	100-41-4	1.0	1.0	120
	Toluene	108-88-3	1.0	1.0	120
	Xylenes (total)	1330-20-7	1.0	1.0	120
Additional 8020A	1,2,4 Trimethylbenzene	95-63-6	1.0	1.0	120
	1,3,5 Trimethylbenzene	108-67-8	1.0	1.0	120
	Acetone	67-64-1	10	10	1200
	MEK (2-butanone)	78-93-3	5.0	5.0	620
	MIBK (4-methyl-2-pentanone)	108-10-1	5.0	5.0	620
	Naphthalene	91-20-3	2.0	2.0	250
	Styrene	100-42-5	1.0	1.0	120
	Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	1.0	120
8021.A	Benzene	71-43-2	1.0	1.0	120
	Bromobenzene	108-86-1	1.0	1.0	120
	Bromochloromethane	74-97-5	1.0	1.0	120
	Bromodichloromethane	75-27-4	1.0	1.0	120
	Bromoform	75-25-2	1.0	1.0	120
	Bromomethane	74-83-9	1.0	1.0	120
	n-butylbenzene	104-51-8	1.0	1.0	120
	sec-Butylbenzene	135-98-8	1.0	1.0	120
	tert-Butylbenzene	98-06-6	1.0	1.0	120
	Carbon Tetrachloride	56-23-5	1.0	1.0	120
	Chlorobenzene	108-90-7	1.0	1.0	120
	Chlorodibromomethane	124-48-1	1.0	1.0	120
	Chloroethane	75-00-3	1.0	1.0	120
	Chloroform	67-66-3	1.0	1.0	120
	Chloromethane	74-87-3	1.0	1.0	120
	2-Chlorotoluene	95-49-8	1.0	1.0	120

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
	4-Chlorotoluene	106-43-4	1.0	1.0	120
	1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0	1.0	120
	1,2-Dibromoethane(EDB)	106-93-4	1.0	1.0	120
	Dibromomethane	74-95-3	1.0	1.0	120
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	120
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	120
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	120
	Dichlorodifluoromethane	75-71-8	1.0	1.0	120
	1,1-Dichloroethane	75-34-3	1.0	1.0	120
	1,2-Dichloroethane	107-06-2	1.0	1.0	120
	1,1-Dichloroethene	75-35-4	1.0	1.0	120
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	120
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	120
	1,2-Dichloropropane	78-87-5	1.0	1.0	120
	1,3-Dichloropropane	142-28-9	1.0	1.0	120
	2,2-Dichloropropane	590-20-7	1.0	1.0	120
	1,1-Dichloropropene	563-58-6	1.0	1.0	120
	cis-1,3-Dichloropropene	10061-01- 5	1.0	1.0	120
	trans-1,3-Dichloropropene	10061-02- 6	1.0	1.0	120
	Ethylbenzene	100-41-4	1.0	1.0	120
	Hexachlorobutadiene	87-68-3	1.0	1.0	120
	Isopropylbenzene	98-82-8	1.0	1.0	120
	p-Isopropyltoluene	99-87-6	1.0	1.0	120
	Methylene Chloride	75-09-2	5.0	5.0	620
	Naphthalene	91-20-3	2.0	2.0	250
	n-Propylbenzene	10306501	1.0	1.0	120
	Styrene	100-42-5	1.0	1.0	120
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	120
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	120
	Tetrachloroethene	127-18-4	1.0	1.0	120
	Toluene	108-88-3	1.0	1.0	120
	1,2,3-Trichlorobenzene	87-61-6	1.0	1.0	120
	1,2,4-Trichlorobenzene	120-82-1	1.0	1.0	120
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	120
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	120

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
	Trichloroethene	79-01-6	1.0	1.0	120
	Trichlorofluoromethane	75-69-4	1.0	1.0	120
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	120
	1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	120
	1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	120
	Vinyl Chloride	75-01-4	1.0	1.0	120
	Xylenes (total)	1330-20-7	1.0	1.0	120

The analytes listed as Additional 8010B and Additional 8020A are amenable to these tests and may be analyzed at client request.

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Table A-2	
Recommended Conditions for Method 8020A	
Parameter	Recommended Conditions
Temperature program	50°C, 1min, 10°C/min to 200°C, 1min
Column 1	Rtx-502.2 or DB-502.2 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 (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3	
Recommended Conditions for Method 8010B or Method 8010B/8020A	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 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, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-4	
Recommended Conditions for Method 8021A	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 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, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocab 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocab 3000)
Transfer line / valve temp	115°C

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Standard	Components	Working Solution ug/mL	Spike amount uL (for 5 mL purge)	Final concentration ug/L (ug/kg)
8020A IS/SS	4-Chlorotoluene (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40		40
8010B IS/SS	4-chlorotoluene (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40		40
8021A IS/SS	Fluorobenzene (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40	10	40

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 for method 8010. 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. Target analyte for 8021.

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 8020. 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

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Conc in 4g sample µg/kg	Conc in purged extract µg/L (100µL/5 mL)
8020A SS	4-chlorotoluene (SS)	20	500	2500	20
8020A IS	1-chloro-4-fluorobenzene	20	5	N/A	20
8010B SS	4-chlorotoluene (SS)	20	500	2500	20
8010B IS	1-chloro-4-fluorobenzene	20	5	N/A	20
8021A SS	Fluorobenzene	20	500	2500	20
	1-4-Dichlorobutane	20	500	2500	20
8021A IS	1-chloro-4-fluorobenzene	20	5	N/A	20

Standard	Components	Working Solution ug/mL	Spike amount uL (5 mL purge)	Final concentration ug/L (ug/kg)
8020A MS	Benzene	10	5	10
	Toluene	10		10
	Chlorobenzene	10		10
8010B MS	Chlorobenzene	10	5	10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10
8021A MS	Benzene	10	5	10
	Toluene	10		10
	Chlorobenzene	10		10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10

**ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A**

Table A-8				
Concentrations for LCS and MS/MSD compounds, medium level soil				
Standard	Components	Working Solution ug/mL	Spike amount uL	Final concentration ug/L (ug/kg)
8020A MS	Benzene	10	500	10
	Toluene	10		10
	Chlorobenzene	10		10
8010B MS	Chlorobenzene	10	500	10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10
8021A MS	Benzene	10	500	10
	Toluene	10		10
	Chlorobenzene	10		10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Table A-9		
Continuing Calibration and Initial Demonstration Limits		
Analyte	Continuing Calibration and Initial Demonstration Recovery Limits, +/- %	Initial Demonstration RSD Limits, %
Method 8010B		
Bromodichloromethane	24.0	21.5
Bromoform	26.5	23.5
Bromomethane	41.5	38
Carbon Tetrachloride	31.5	28
Chlorobenzene	28.0	25
Chloroethane	23.0	22
2-Chloroethyl vinyl ether	40.0	41.5
Chloroform	25.0	22.5
Chloromethane	40.5	37
Dibromochloromethane	34.5	31.5
1,2-Dichlorobenzene	30.0	27.5
1,3-Dichlorobenzene	50.5	45.5
1,4-Dichlorobenzene	30.5	27.5
1,1-Dichloroethane	16.0	16
1,2-Dichloroethane	28.5	26
1,1-Dichloroethene	37.0	33
trans-1,2-Dichloroethene	36.0	32
Dichloromethane	22.5	20
1,2-Dichloropropane	26.0	26
cis-1,3-Dichloropropene	36.0	36.5
trans-1,3-Dichloropropene	36.0	36.5
1,1,2,2-Tetrachloroethane	51.0	46
Tetrachloroethene	30.0	27
1,1,1-Trichloroethane	29.0	24.5
1,1,2-Trichloroethane	21.5	19.5
Trichloroethene	23.0	21
Trichlorofluoromethane	33.5	30
Vinyl chloride	31.5	28.5
Additional compounds	35	25

ANALYSIS OF VOLATILE ORGANICS BASED ON
METHODS 8010B, 8020A, AND 8021A

Revision No: 2.1

Revision Date: 08/12/96

Page A22 of A21

Table A-10		
Continuing Calibration and Initial Demonstration Limits		
Analyte	Continuing Calibration and Initial Demonstration Recovery Limits, +/- %	Initial Demonstration RSD Limits, %
Method 8020A		
Benzene	23.0	20.5
Chlorobenzene	19.5	17.5
1,2-Dichlorobenzene	32.0	29.0
1,3-Dichlorobenzene	27.5	25.0
1,4-Dichlorobenzene	30.5	27.5
Ethylbenzene	37.0	33.5
Toluene	22.5	20.0
Additional compounds	35.0	25.0

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8000A is applied to the analysis of organochlorine pesticides and Aroclors (PCBs) by GC/ECD. This Appendix is to be applied when 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.

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 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 8000A 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 8000A 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the 8000A 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 8000A 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. Dibutylchloroendate may be used at client request. Refer to tables B-5, B-6, and B-7 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 8000A section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000A section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000A section of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method.

10.3. 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 1701 column.

10.4. Column Degradation Evaluation

Before any calibration runs, either initial or daily, the column evaluation mix must be injected. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 20% 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.

10.5. Initial Calibration

Refer to Section 10 of the 8000A section of this SOP for details of calibration procedures.

10.5.1. Refer to Table B-8 for the initial calibration analytical sequence.

10.5.2. The response for each single-peak analyte will be calculated by the external standard procedure described in the general method for GC analysis.

10.5.3. The surrogate calibration curve is calculated from the Individual AB mix (or from the Aroclor 1016/1260 mix if the analysis is for acid cleaned

Aroclor only). Surrogates in the other calibration standards are used only as retention time markers.

10.5.4. For multi-component pesticides and Aroclors:

Two options are possible for quantitation of multicomponent pesticides and Aroclors. The same quantitation option must be used for standards and samples.

10.5.4.1. 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 multi-peak pesticide or 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.)

10.5.4.2. 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.

10.5.5. For multicomponent analytes, the low level standard must be analyzed as part of the initial calibration. If multicomponent analytes are present above half the reporting limit in any of the samples, these samples must be reanalyzed and quantitated against a valid five point calibration (unless a 5 point calibration for multicomponent was included as part of the initial calibration).

10.5.6. The analyst may include a full 5 point calibration for any of the multicomponent analytes with the initial calibration.

10.5.7. For Aroclor only analysis, a five point calibration of the 1016/1260 mix is generated with at least low level single points for the other Aroclor mixes. If multicomponent analytes are present above half the reporting limit in any of the samples, these samples must be reanalyzed and quantitated against a valid five point calibration.

10.6. Daily Calibration Verification

The daily calibration verification must be analyzed within 24 hours of the start of the initial calibration and at least once every 24 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

- 10.6.1. At a minimum, the daily calibration includes analysis of the breakdown mix followed by mid level standards of any single component analytes. (The individual mix AB and any additional single component analytes.)
- 10.6.2. If any multicomponent analytes are to be quantitated, then the daily calibration must include mid level standards of the appropriate multicomponents.
- 10.6.3. If multicomponent analytes do not have a full five point initial calibration or are not included with the daily calibration, then any multicomponent analytes found at greater than half the reporting limit must be reanalyzed and quantitated against a five point. Note that, at a minimum, all multicomponent analytes of interest must have a low level standard analyzed as part of the initial calibration to ensure that the sensitivity of the instrument is sufficient for pattern recognition.
- 10.6.4. Multicomponent analytes may be quantitated if there is a valid 5 point initial calibration on the instrument for the multicomponent analyte and a mid level standard of the multicomponent is analyzed within 24 hours and prior to the sample. The mid level standard must meet the 15% calibration criteria for quantitation.
- 10.6.5. The retention time windows for any analytes included in the daily calibration are updated.

10.7. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration standard. This is analyzed after every 10 samples, including matrix spikes, LCS, and method blanks. The continuing calibration standard need not include multicomponent analytes.

- 10.7.1. The level 3 calibration standard is used for the continuing calibration.

- 10.7.2. If only Aroclors are requested, the continuing calibration standard will consist of the mid level 1016/1260 Aroclor standard with the surrogates added.

11. PROCEDURE

- 11.1. Refer to the method 8000A 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 8000A 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.

- 12.2.1. The chromatogram is evaluated for technical chlordane if alpha and gamma chlordane are present. If technical chlordane is present it must be quantitated against a 5 point technical chlordane curve.

12.3. Quantitation of Multicomponent Analytes

Use 3-10 major peaks or total area for quantitation as described in section 10.5.4. initial calibration of multicomponent analytes.

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.

- 12.3.1. 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.
- 12.4. Second column confirmation of Aroclors and other 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 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.
- 12.6. Calculation of Column Degradation/% Breakdown (%B)

Equation 15

$$DDT \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

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.

Equation 16

$$Endrin \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_E} \times 100$$

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 8000A section of this SOP.

15. WASTE MANAGEMENT

- 15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update II, September 1994, Method 8080A

17. MISCELLANEOUS**17.1. Modifications from Reference Method**

- 17.1.1. Standards are made up in hexane, not isooctane. Use of standards in the same solvent as the sample extracts (hexane) ensures that analysis of the standard is consistent with analysis of the sample.
- 17.1.2. Only a single point initial calibration is required for multicomponent analytes (Aroclors, toxaphene and technical chlordane). However, if any of these analytes are found, they must be quantitated against a valid 5 point initial calibration.
- 17.1.3. Endrin ketone, alpha-chlordane and gamma-chlordane are included in the calibration standards. These are not standard 8080A analytes.

17.2. Modifications from Previous Revisions

None

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.

17.3.1. *Refer to the SOP change form on file in North Canton's Quality Assurance office.*

17.4. Tables

Table B-1			
Standard Analyte list and Reporting Limits			
Compound	Reporting Limit, ug/L or ug/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
a-BHC	0.05	1.7	50
b-BHC	0.05	1.7	50
d-BHC	0.05	1.7	50
g-BHC (Lindane)	0.05	1.7	50
Chlordane (technical)	0.5	17	500
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	0.1	3.3	100
Toxaphene	2.0	67	2000
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
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzillate	0.1	3.3	100
Kepone	1.0	33	1000

Note: alpha chlordane, gamma chlordane, and endrin ketone are not 8080A compounds.

**ANALYSIS OF ORGANOCHLORINE PESTICIDES AND PCBs
BASED ON METHOD 8080A**

The following concentration factors are assumed in calculating the Reporting Limits:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>
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	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	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

ANALYSIS OF ORGANOCHLORINE PESTICIDES AND PCBs
BASED ON METHOD 8080A

Table B-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6 ²
Individual Mix AB¹						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
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
b-BHC	5	10	25	50	100	200
d-BHC	5	10	25	50	100	200
a-BHC	5	10	25	50	100	200
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
a-Chlordane	5	10	25	50	100	200
g-Chlordane ³	5	10	25	50	100	200
Multicomponent Standards						
Chlordane (Technical)	50	100	250	500	1000	2000
Toxaphene	200	400	1000	2000	4000	8000
Aroclor 1016/1260	100	200	500	1000	2000	4000
Aroclor 1242	100	200	500	1000	2000	4000
Aroclor 1221 +1254	100	200	500	1000	2000	4000
Aroclor 1232	100	200	500	1000	2000	4000
Aroclor 1248	100	200	500	1000	2000	4000
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl ⁴	5	10	25	50	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 are not 8080A target compounds but may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

⁴ Dibutylchlorendate may be included as a surrogate at the same concentration as decachlorobiphenyl if needed for specific client requests.

ANALYSIS OF ORGANOCHLORINE PESTICIDES AND PCBs
BASED ON METHOD 8080A

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 ug/L or ug/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	6.67	200
Aldrin	0.20	6.67	200
Heptachlor	0.20	6.67	200
Dieldrin	0.50	16.7	500
Endrin	0.50	16.7	500
4,4' DDT	0.50	16.7	500
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table B-6			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup ug/L or ug/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10.000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table B-7		
LCS/Matrix Spike and Surrogate Spike levels for TCLP ug/L or ug/kg		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

**ANALYSIS OF ORGANOCHLORINE PESTICIDES AND PCBs
BASED ON METHOD 8080A**

**Table B-8
Suggested Analytical Sequence**

Initial Calibration

Injection #

1	Solvent blank (optional)	
2	Breakdown Mix	
3-7	Individual mix AB	All levels
8	Aroclor 1016/1260	Level 1 ¹
9	Aroclor 1232	Level 1 ¹
10	Aroclor 1242	Level 1 ¹
11	Aroclor 1248	Level 1 ¹
12	Aroclor 1221/1254	Level 1 ¹
13	Technical Chlordane	Level 1 ¹
14	Toxaphene	Level 1 ¹
15	Solvent blank	
16-25	Sample 1-10	
	Solvent blank (optional)	
26	Individual mix AB	Mid level (Continuing calibration)
etc	Sample 11-20	
	Solvent blank (optional)	
	Individual mix AB	Mid level (Continuing calibration)
	Sample 21-30	
	After 24 hours:	
	Breakdown mix	
	Individual mix AB	
	Any other single component analytes	
	Any multicomponent analytes for quantitation	

¹ A five point curve for any of the multicomponent analytes may be included, and is required for quantitation.

Daily Calibration

At least every 24 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.

- All samples that have a positive hit for a multicomponent must be run after and within 24 hours of the standard that contains that multicomponent. The standard mix that contains this multicomponent must meet all the calibration criteria.

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
Aroclor 1016 + 1260	62.5-110	20

**ANALYSIS OF PHENOXY ACID HERBICIDES BASED
ON METHOD 8150B or 8151**

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 C1 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 8000A 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 8000A SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the 8000A section of this SOP. A Ni₆₃ electron capture detector is required.
- 6.2. Refer to Table C2 for analytical columns.
- 6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

**ANALYSIS OF PHENOXY ACID HERBICIDES BASED
ON METHOD 8150B or 8151****7. REAGENTS AND STANDARDS**

- 7.1. Refer to section 7 of the 8000A section of this SOP for general information on reagents and standards.
- 7.2. Refer to Table C-3 and C-4 for details of calibration and other standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000A section of this SOP.

9. QUALITY CONTROL

- 9.1. Refer to Section 9 of the 8000A 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 C-6 for minimum performance criteria for the initial demonstration of capability.
- 9.3. Refer to Table C-5 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Refer to Section 10 of the 8000A section of this SOP for general calibration requirements.
- 10.2. External standard calibration is used for this method.
- 10.3. The level 3 calibration standard is used for the continuing calibration.
- 10.4. Refer to Table C-2. for details of GC operating conditions.

11. PROCEDURE

- 11.1. Refer to the method 8000A section of this SOP for procedural requirements.
- 11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001.

**ANALYSIS OF PHENOXY ACID HERBICIDES BASED
ON METHOD 8150B or 8151**

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 24 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 24 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 10 samples (including LCS, method blank, and MS samples), a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. It is not necessary but it is permissible to update retention time windows with continuing calibrations.

11.5. Gas Chromatography

Chromatographic conditions are listed in Table C-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000A 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 20ug/L calibration standard for 2,4-D contains 21.3 ug/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 C-5 lists minimum performance standards required by Quanterra for the four replicate initial demonstration or capability (required by Section 13.2 of the 8000A 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 according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Method 8150B, SW-846. Update II, September 1994

Method 8151, SW-846. Update II, September 1994

17. MISCELLANEOUS**17.1. Modifications from Reference Method**

Refer to the method 8000A section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

This procedure can now be applied to analysis based on method 8151A.

17.3. Tables

Standard Analyte list				
Compound	CAS Number	Reporting Limit, ug/L or ug/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:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>	<u>Dilution Factor</u>
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 C-1.

ANALYSIS OF PHENOXY ACID HERBICIDES BASED
ON METHOD 8150B or 8151

Table C-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	2uL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table C-1.

ANALYSIS OF PHENOXY ACID HERBICIDES BASED
ON METHOD 8150B or 8151

Table C-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 (34,000)	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

¹The 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

The reporting limits listed in Table C-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.

	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.

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
MCPD	50-150	25

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8000A is applied to the analysis of organochlorine pesticides and Aroclors (PCBs) by GC/ECD. This Appendix is to be applied when SW-846 Method 8081 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 D-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.

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 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 8000A 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 8000A 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the 8000A section of this SOP. A ⁶³Ni electron capture detector is required.
- 6.2. Refer to Table D-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 8000A section of this SOP for general requirements for reagents and supplies. All standards for this method must be replaced
- 7.2. Refer to Table D-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 tables D-5, D-6, and D-7 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 D-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000A section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000A section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000A section of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method.

10.3. Refer to Table D-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table D-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 1701 column.

10.4. Column Degradation Evaluation

Before any calibration runs, either initial or daily, the column evaluation mix must be injected. 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.

10.5. Initial Calibration

Refer to Section 10 of the 8000A section of this SOP for details of calibration procedures.

10.5.1 Refer to Table D-8 for the initial calibration analytical sequence.

- 10.5.2. The response for each single-peak analyte will be calculated by the external standard procedure described in the general method for GC analysis.
- 10.5.3. The surrogate calibration curve is calculated from the Individual AB mix (or from the Aroclor 1016/1260 mix if the analysis is for acid cleaned Aroclor only). Surrogates in the other calibration standards are used only as retention time markers.
- 10.5.4. For multi-component pesticides and Aroclors:

Two options are possible for quantitation of multicomponent pesticides and Aroclors. The same quantitation option must be used for standards and samples.

10.5.4.1. 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 multi-peak pesticide or 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.)

10.5.4.2. 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.

- 10.5.5. For multicomponent analytes, the low level standard must be analyzed as part of the initial calibration. If multicomponent analytes are present above half the reporting limit in any of the samples, these samples must be reanalyzed and quantitated against a valid five point calibration (unless a 5 point calibration for multicomponent was included as part of the initial calibration).

- 10.5.6. The analyst may include a full 5 point calibration for any of the multicomponent analytes with the initial calibration.
- 10.5.7. For Aroclor only analysis, a five point calibration of the 1016/1260 mix is generated with at least low level single points for the other Aroclor mixes. If multicomponent analytes are present above half the reporting limit in any of the samples, these samples must be reanalyzed and quantitated against a valid five point calibration.

10.6. Daily Calibration Verification

The daily 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 daily calibration.

- 10.6.1. At a minimum, the daily calibration includes analysis of the breakdown mix followed by mid level standards of any single component analytes. (The individual mix AB and any additional single component analytes.)
- 10.6.2. If any multicomponent analytes are to be quantitated, then the daily calibration must include mid level standards of the appropriate multicomponents. For the Aroclors, it is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. However, if any other Aroclors are detected the samples must be reinjected and bracketed with daily calibration standards of the appropriate Aroclor. If a specific Aroclor is expected, it should be included in the daily calibration check.
- 10.6.3. If multicomponent analytes do not have a full five point initial calibration or are not included with the daily calibration, then any multicomponent analytes found at greater than half the reporting limit must be reanalyzed and quantitated against a five point. Note that, at a minimum, all multicomponent analytes of interest must have a low level standard analyzed as part of the initial calibration to ensure that the sensitivity of the instrument is sufficient for pattern recognition.

**ANALYSIS OF ORGANOCHLORINE PESTICIDES AND PCBs
ON METHOD 8081**

10.6.4. Multicomponent analytes may be quantitated if there is a valid 5 point initial calibration on the instrument for the multicomponent analyte and a mid level standard of the multicomponent is analyzed within 12 hours and prior to the sample. The mid level standard must meet the 15% calibration criteria for quantitation.

10.6.5. The retention time windows for any analytes included in the daily calibration are updated.

10.7. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration 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 continuing calibrations more frequently in order to minimize reruns.) The continuing calibration standard need not include multicomponent analytes.

10.7.1. The level 3 calibration standard is used for the continuing calibration.

10.7.2. If only Aroclors are requested, the continuing calibration standard will consist of the mid level 1016/1260 Aroclor standard with the surrogates added.

10.7.3. For method 8081, if the bracketing standard is out of control, all samples injected after the last standard that met the continuing calibration criteria must be re-injected.

11. PROCEDURE

11.1. Refer to the method 8000A 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 D-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table D-8.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000A 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.

12.2.1. The chromatogram is evaluated for technical chlordane if alpha and gamma chlordane are present. If technical chlordane is present it must be quantitated against a 5 point technical chlordane curve.

12.3. Quantitation of Multicomponent Analytes

Use 3-10 major peaks or total area for quantitation as described in section 10.5.4. initial calibration of multicomponent analytes.

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.

12.3.1. 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.

12.4. Second column confirmation of Aroclors and other 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 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.

12.6. Calculation of Column Degradation/% Breakdown (%B)

Equation 17

$$DDT \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

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.

Equation 18

$$Endrin \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_E} \times 100$$

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 recovery of 80-120% for standard analytes. These limits may not be achievable for certain analytes such as kepone and dichlone. The laboratory must develop internal performance limits for these analytes. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000A section of this SOP.

15. WASTE MANAGEMENT

- 15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update II, September 1994, Method 8081

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. The recommended limits of 80-120% for the QC reference sample listed in the reference method are not appropriate at the lower concentrations determined in this SOP. Historical limits will be used for LCS control.

17.1.2. Decachlorobiphenyl and tetrachloro-m-xylene are used as surrogates for both the dual and single column methods.

17.2. Modifications from Previous Revisions

The 8081 procedure was added to this revision.

17.3. Tables

Table D-1 Standard Analyte list and Reporting Limits ...			
Compound	Reporting Limit, ug/L or ug/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
a-BHC	0.05	1.7	50
b-BHC	0.05	1.7	50
d-BHC	0.05	1.7	50
g-BHC (Lindane)	0.05	1.7	50
αChlordane (technical)	0.05	1.7	50
γChlordane (technical)	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	0.1	3.3	100
Toxaphene	2.0	67	2000
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
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzillate	0.1	3.3	100
Kepone	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table D-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	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table D-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6 ²
Individual Mix AB¹						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
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
b-BHC	5	10	25	50	100	200
d-BHC	5	10	25	50	100	200
a-BHC	5	10	25	50	100	200
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
a-Chlordane	5	10	25	50	100	200
g-Chlordane	5	10	25	50	100	200
Multicomponent Standards						
Chlordane (Technical)	50	100	250	500	1000	2000
Toxaphene	200	400	1000	2000	4000	8000
Aroclor 1016/1260	100	200	500	1000	2000	4000
Aroclor 1242	100	200	500	1000	2000	4000
Aroclor 1221 +1254	100	200	500	1000	2000	4000
Aroclor 1232	100	200	500	1000	2000	4000
Aroclor 1248	100	200	500	1000	2000	4000
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	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.

Table D-4	
Column Degradation Evaluation Mix ng/mL	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table D-5			
LCS/Matrix Spike and Surrogate Spike levels ug/L or ug/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	6.67	200
Aldrin	0.20	6.67	200
Heptachlor	0.20	6.67	200
Dieldrin	0.50	16.7	500
Endrin	0.50	16.7	500
4,4'-DDT	0.50	16.7	500
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table D-6			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup ug/L or ug/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table D-7		
LCS/Matrix Spike and Surrogate Spike levels for TCLP ug/L or ug/kg		
	Aqueous	Waste
Heptachlor	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table D-8
Suggested Analytical Sequence

Initial Calibration

Injection

1	Solvent blank (optional)	
2	Breakdown Mix	
3-7	Individual mix AB	All levels
8	Aroclor 1016/1260	Level 1 ¹
9	Aroclor 1232	Level 1 ¹
10	Aroclor 1242	Level 1 ¹
11	Aroclor 1248	Level 1 ¹
12	Aroclor 1221/1254	Level 1 ¹
13	Technical Chlordane	Level 1 ¹
14	Toxaphene	Level 1 ¹
15	Solvent blank	
16-35	Sample 1-20	
	Solvent blank (optional)	
36	Individual mix AB	Mid level (Continuing calibration)
etc	Samples	
	After 24 hours:	
	Breakdown mix	
	Individual mix AB	
	Any other single component analytes	
	Any multicomponent analytes for quantitation	

¹ A five point curve for any of the multicomponent analytes may be included, and is required for quantitation.

Daily 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.

- All samples that have a positive hit for a multicomponent must be run after and within 24 hours of the standard that contains that multicomponent. The standard mix that contains this multicomponent must meet all the calibration criteria.

Controlled Copy
Copy No. _____

UNCONTROLLED COPY

SOP No. CORP-MT-0007NC
Revision No. 1.1
Revision Date: 04-17-97
Page: 1 of 40

QUANTERRA STANDARD OPERATING PROCEDURE

**TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY
COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, SW846 7471A AND MCAWW
245.5
(SUPERSEDES: REVISION 1)**

Prepared by: Susan Palmer 4/23/97

Reviewed by: Roger K. Soth
Technology Specialist

Approved by: Galvin Park 4/22/97
Quality Assurance Manager

Approved by: Steve J. Jack 4-23-97
Environmental Health and Safety Coordinator

Approved by: Christine K. Opus 4-23-97
Laboratory Director

Approved by: Bill B. Huff
Corporate Quality Assurance or Technology

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD	3
3. DEFINITIONS	3
4. INTERFERENCES	3
5. SAFETY	5
6. EQUIPMENT AND SUPPLIES.....	6
7. REAGENTS AND STANDARDS.....	7
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE.....	9
9. QUALITY CONTROL.....	9
10. CALIBRATION AND STANDARDIZATION.....	13
11. PROCEDURE	14
12. DATA ANALYSIS AND CALCULATIONS.....	18
13. METHOD PERFORMANCE.....	20
14. POLLUTION PREVENTION.....	20
15. WASTE MANAGEMENT.....	20
16. REFERENCES.....	21
17. MISCELLANEOUS (TABLES, APPENDICES. ETC. . . .).....	21

LIST OF APPENDICES:

APPENDIX A - TABLES	27
APPENDIX B - QUANTERRA Hg DATA REVIEW CHECKLIST	31
APPENDIX C - MSA GUIDANCE	33
APPENDIX D - TROUBLESHOOTING GUIDE.....	36
APPENDIX E- CONTAMINATION CONTROL GUIDELINES.....	38
APPENDIX F - PREVENTATIVE MAINTENANCE.....	40

1. SCOPE AND APPLICATION

- 1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A and MCAWW Method 245.5.
- 1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.
- 1.3. Methods 7471A and 245.5 are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits and sludge-type materials. All matrices require sample preparation prior to analysis.
- 1.4. The Quanterra reporting limit for mercury in solid matrices is 0.1 mg/kg based a 0.2 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

- 2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

- 3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

- 4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.
- 4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.
- 4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

- 4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.
- 4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.
- 4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

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. The following specific hazards are known:
 - 5.3.1. The following materials are known to be **corrosive**:
hydrochloric acid, nitric acid and sulfuric acid.
 - 5.3.2. The following materials are known to be **oxidizing agents**:
nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.
 - 5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:
 - 5.3.3.1. Equal volumes of 0.1 M KMnO_4 and 10% H_2SO_4 , or
 - 5.3.3.2. Iodine, 0.25% in a 3% KI solution.
 - 5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).
- 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. 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 with the sash closed as far as the operation will permit.
- 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. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.
- 5.8. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.
- 5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

- 6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C).
- 6.2. Atomic Absorption Spectrophotometer equipped with:
 - 6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.
 - 6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).
 - 6.2.3. Peristaltic pump which can deliver 1 L/min air.
 - 6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.
 - 6.2.5. Recorder or Printer.

- 6.2.6. Aeration Tubing: A straight glass frit having a coarse porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.
- 6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that achieve the same purpose are also acceptable (i.e., Gortex filter).

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

- 6.3. BOD bottles or equivalent.
- 6.4. Nitrogen or argon gas supply, welding grade or equivalent.
- 6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.
- 6.6. Class A volumetric flasks.
- 6.7. Top-loading balance, capable of reading up to two decimal places.
- 6.8. Thermometer (capable of accurate readings at 95 °C).
- 6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

- 7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
- 7.2. Stock (1000 ppm) mercury standards (in 10% HNO₃) are purchased as custom Quanterra solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

- 7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO₃. This acid (2 mL of concentrated HNO₃) must be added to the flask/bottle before the addition of the stock standard aliquot.
- 7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO₃. This acid (150 uL of concentrated HNO₃) must be added to the flask/bottle before the addition of the stock standard aliquot.
- 7.5. The calibration standards must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample prep bottles and proceeding as specified in Section 11.1

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

- 7.6. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.
- 7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.
- 7.8. Nitric acid (HNO₃), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.
- 7.9. Sulfuric acid (H₂SO₄), concentrated, trace metal grade or better.
 - 7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H₂SO₄ to 1 liter with reagent water.
- 7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

- 7.11. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃.
- 7.12. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

- 7.13. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

- 7.14. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.
- 7.15. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.
- 8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

- 9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A or the 245.5, the following requirements must be met.

- 9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any 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. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a 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 pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS) are not included in the sample count for determining the size of a preparation batch. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.

9.4. 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 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 20 times higher than the blank contamination level).

- 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.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. 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. The LCS must be carried through the entire analytical procedure. The CCV results can be reported as the LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.
- In the instance where the LCS recovery is > 120% 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 case narrative.”
- 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.
- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. 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/MSD's. 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 Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD 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 reparation 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 4 times 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.
- 9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include reparation of the associated samples.
- 9.8. 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 must be a mid-range standard at a concentration other than that of

the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

- 9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. 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 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.
- 10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.
- 10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.
- 10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.
- 10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the Quanterra reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

- 10.6. The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.
- 10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Standard and Sample Preparation:

- 11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.
- 11.1.2. Transfer 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working standard (7.5) into a series of sample digestion bottles.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.
- 11.1.3. Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under 11.1.5 or 11.1.6 below.
- 11.1.4. Transfer triplicate 0.2 g portions of a well mixed sample into a clean sample digestion bottle. Continue preparation as described under 11.2.2 or 11.2.3 below.
- 11.1.5. Water Bath protocol:
 - 11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.
To each **sample** bottle: Add 10 mL of reagent water and 5 mL of aqua regia.
 - 11.1.5.2. Heat for 2 minutes in a water bath at 90 - 95 ° C.
 - 11.1.5.3. Cool.
 - 11.1.5.4. Add 40 mL of distilled water.
 - 11.1.5.5. Add 15 mL of potassium permanganate solution.

- 11.1.5.6. Heat for 30 minutes in the water bath at 90 - 95 °C.
- 11.1.5.7. Cool.
- 11.1.5.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.
- 11.1.5.9. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add 50 mL of reagent water.
- 11.1.5.10. Continue as described under Section 11.2.

11.2. Sample Analysis:

- 11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.
- 11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.
- 11.2.3. Manual determination:
 - 11.2.3.1. Treating each sample individually, purge the head space of the sample bottle for at least one minute.
 - 11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.
 - 11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.
 - 11.2.3.4. Place the aeration device into 100 mLs of 1% HNO₃ and allow to bubble rinse until the next sample is analyzed.

- 11.2.4. Automated determination: Follow instructions provided by instrument manufacturer.
- 11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.
- 11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.
- 11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.
- 11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.
- 11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.
- 11.2.10. The following analytical sequence must be used with 7471A and 245.5:

Instrument Calibration

ICV

ICB

Maximum 10 samples

CCV

CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run

CCV

CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7471A and 245.5.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

- 11.2.11. The following run sequence is consistent with 7471A, CLP and 245.5 and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration

ICV

ICB

CRA*

CCV

CCB

10 samples

CCV

CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.

CCV

CCB

Refer to the appropriate CLP SOP (CORP-MT-0008) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

- 11.2.12. 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.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

- 11.3. 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.

- 11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.
- 11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- 11.6. 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:

$$\%R = 100 \left(\frac{\text{Found(ICV)}}{\text{True(ICV)}} \right)$$

- 12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \left(\frac{\text{Found(CCV)}}{\text{True(CCV)}} \right)$$

- 12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100 \left(\frac{\text{SSR} - \text{SR}}{\text{SA}} \right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

- 12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100 \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

$$RPD = 100 \left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2} \right)} \right]$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

- 12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$\text{mg/kg. dry weight} = (C \times V \times D) / (W \times S)$$

Where:

C = Concentration (ug/L) from instrument readout

V = Volume of digestate (L)

D = Instrument dilution factor

W = Weight in g 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 a wet weight basis, the "S" factor should be omitted from the above equation.

- 12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$\text{mg/kg. dry weight} = (C) / (W \times S)$$

Where:

C = Concentration (ug) from instrument readout

W = Weight in g of wet sample digested

$$S = \text{Percent solids}/100$$

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 a wet weight basis, the "S" factor should be omitted from the above equation.

- 12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \left(\frac{\text{Found}(LCS)}{\text{True}(LCS)} \right)$$

- 12.8. 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. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The laboratory control sample should recover within 20% of the true value until in house limits are established.

- 13.3. Training Qualification:

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 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 according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

- 16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).
- 16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.5.
- 16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.
- 16.4. QA-003, Quanterra QC Program.
- 16.5. QA-004, Rounding and Significant Figures.
- 16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

- 17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7471A and 245.5.

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

17.1.2.1. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

- 17.1.2.2. 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.
- 17.1.2.3. Method 7471A does not state control criteria within the text of the method. The QC section of 7471A refers the analyst to Section 8.0 of Method 7000A, the generic atomic absorption method, which discusses flame and furnace methods. The ICV criteria stated in Method 7000A is $\pm 10\%$. This SOP requires ICV control limits of $\pm 20\%$ based on the fact that the mercury ICV, unlike the ICV for the flame and furnace analytes, is digested and therefore is equivalent to a LCS. The CLP protocol 245.5 CLP-M recognizes this factor and requires control limits of $\pm 20\%$.

17.1.3. Modifications from 245.5

17.1.3.1. Method 245.5 Section 9.3 states concentrations should be reported as follows: Between 0.1 and 1 ug/g, to the nearest 0.01 ug; between 1 and 10 ug/g, to the nearest 0.1ug; above 10 ug/g, to the nearest ug. Quanterra reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

None.

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. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.

Non-conformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Water Bath Procedure

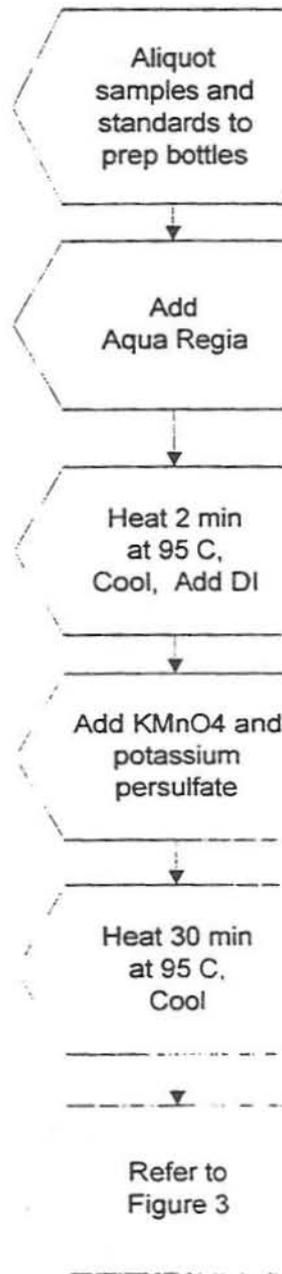
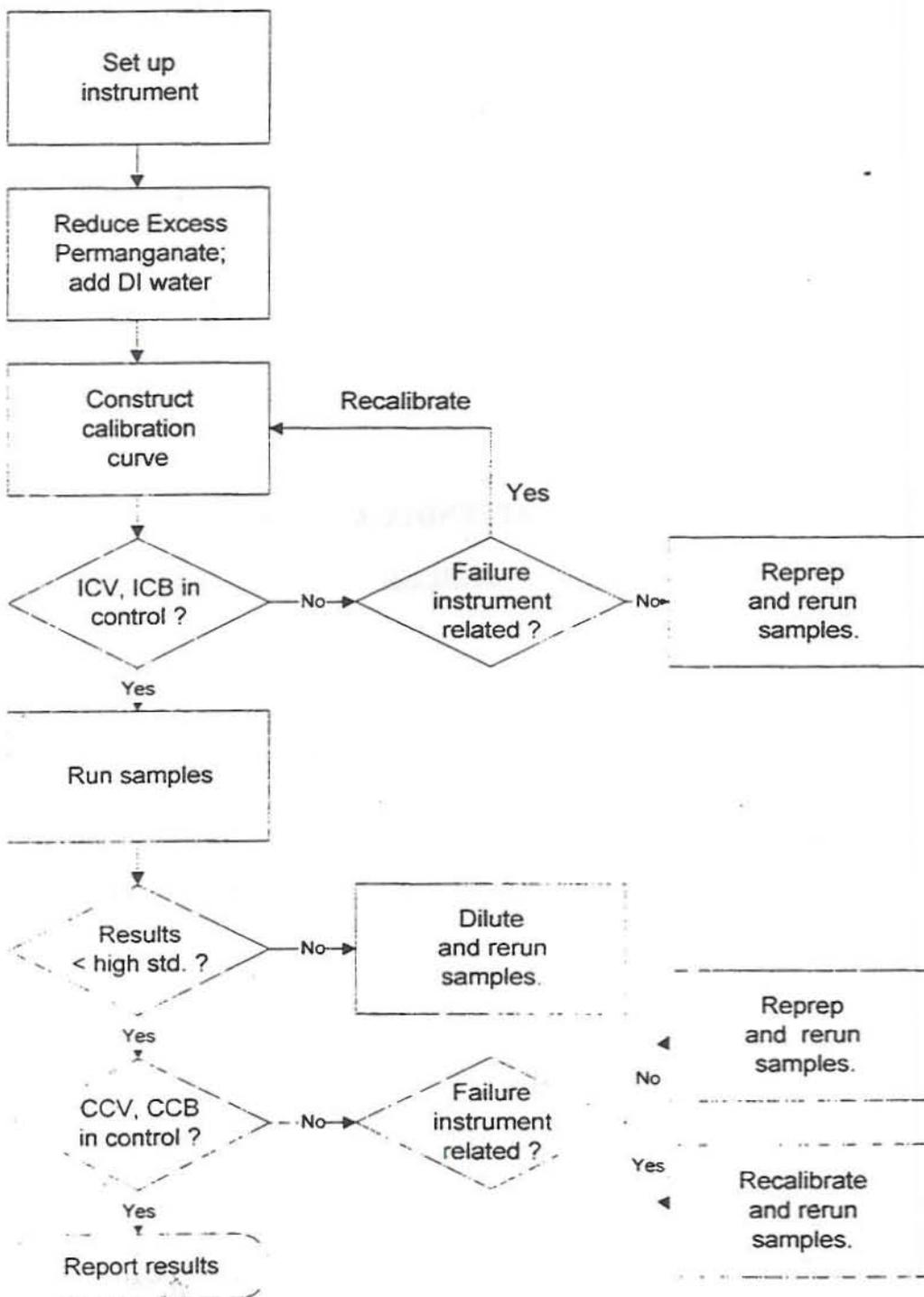


Figure 2. CVAA Mercury Analysis



APPENDIX A
TABLES

**TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC
 STANDARD AND SPIKING LEVELS**

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.002
Std 5 (mg/L)	0.005
Std 6 (mg/L) **	0.010
ICV (mg/L)	0.001 or 0.0025 ***
CCV/LCS (mg/L)	0.0025 or 0.005 ***
MS (mg/L)	0.001

- * SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.
- ** Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.
- *** Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.8).
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 or reprep batch (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
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.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD \leq 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
QUANTERRA Hg DATA REVIEW CHECKLIST

Quanterra Hg Data Review Checklist

Run/Project Information

Run Date: _____ Analyst: _____ Instrument: _____
 Prep Batches Run: _____

Circle Methods used: 7470A / 245.1 : CORP-MT-0005 Rev 1 7471 / 245.5 : CORP-MT-0007 Rev 1
 CLP - AQ : CORP-MT-0006 Rev 0 CLP - SOL : CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard 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?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ Date: _____
 Comments: _____

2nd Level Reviewer : _____ Date: _____

APPENDIX C
MSA GUIDANCE

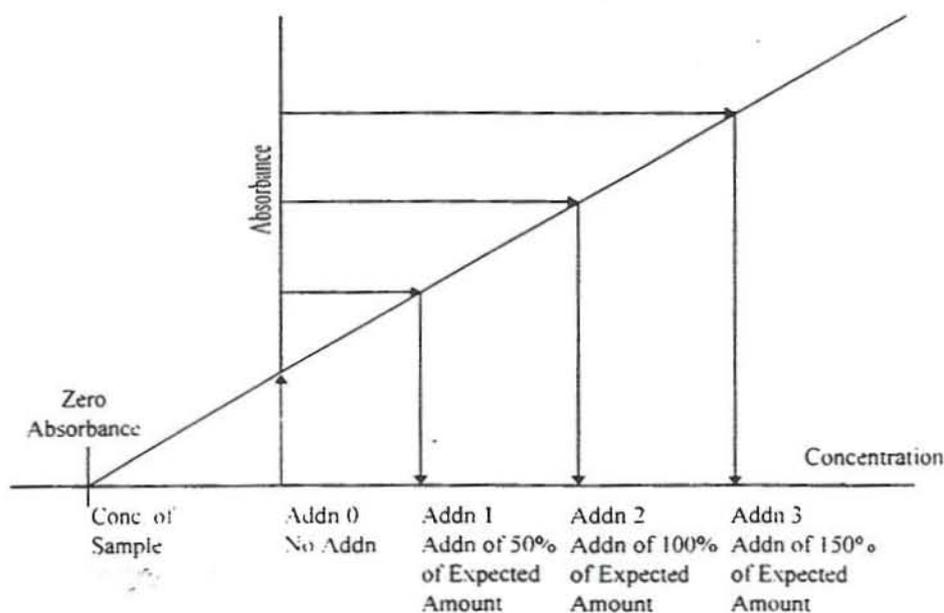
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x -intercept (where $y=0$) of the curve. The concentration in the digestate is equal to the negative x -intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Problem	Cause	Solution
Low sensitivity	Low concentration of standard Sample matrix effects Inadequate digestion Inadequate extraction Inadequate purge gas flow Inadequate carrier gas flow Inadequate chiller temperature Inadequate detector gain Inadequate lamp current Inadequate lamp warm-up time	Check standard concentration Check sample matrix Check digestion procedure Check extraction procedure Check purge gas flow rate Check carrier gas flow rate Check chiller temperature Check detector gain Check lamp current Check lamp warm-up time
High sensitivity	High concentration of standard Sample matrix effects Excessive digestion Excessive extraction Excessive purge gas flow Excessive carrier gas flow Excessive chiller temperature Excessive detector gain Excessive lamp current Excessive lamp warm-up time	Check standard concentration Check sample matrix Check digestion procedure Check extraction procedure Check purge gas flow rate Check carrier gas flow rate Check chiller temperature Check detector gain Check lamp current Check lamp warm-up time
High background	Sample matrix effects Inadequate digestion Inadequate extraction Inadequate purge gas flow Inadequate carrier gas flow Inadequate chiller temperature Inadequate detector gain Inadequate lamp current Inadequate lamp warm-up time	Check sample matrix Check digestion procedure Check extraction procedure Check purge gas flow rate Check carrier gas flow rate Check chiller temperature Check detector gain Check lamp current Check lamp warm-up time
Low background	Excessive digestion Excessive extraction Excessive purge gas flow Excessive carrier gas flow Excessive chiller temperature Excessive detector gain Excessive lamp current Excessive lamp warm-up time	Check digestion procedure Check extraction procedure Check purge gas flow rate Check carrier gas flow rate Check chiller temperature Check detector gain Check lamp current Check lamp warm-up time

APPENDIX D
TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E
CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

PREPARATION AND ANALYSIS OF MERCURY IN SOLID
SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW-846
METHOD 7471A and MCAWW METHOD 245.5
APPENDIX F - PREVENTIVE MAINTENANCE

SOP No. CORP-MT-0007NC
Revision No. 1.1
Revision Date: 04-17-97
Page: 39 of 40

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. 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.

Cold Vapor Atomic Absorption (Leeman PS 200)⁽¹⁾

Daily	Semi-annually	Annually
Clean lens.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000)⁽¹⁾

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

Controlled Copy **CONTROLLED COPY**
Copy No: _____

SCF No. CORP-MS-0002
Revision No. 2
Revision Date: 12/15/97
Implementation Date: 03/15/99
Page: 1 of 66

QUANTERRA STANDARD OPERATING PROCEDURE

**TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON
METHOD 8260B, 624 AND 524.2**

(SUPERSEDES REVISION 1)

Prepared by: Richard Burrows

Reviewed by: *W. B.*
Technology Standardization Committee

Approved by: *[Signature]*
Director, Quality Assurance

Approved by: *Donald P. Dineen*
Director, Environmental Health and Safety

Approved by: *[Signature]*
Manager

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

SOP REQUEST FORM

Client/Project: Louisville District - ARMY

Client Contact: Samir A. Mansy

Client Address: Louisville, KY

Reason Requested: Louisville District Proposal

Requested By: (P.M.) Cathy Knudsen/Stacy Aubuchon

Date Requested: 03/03/99 Date Needed: 03/11/99 (mailed)

No. of Copies: 1 Stapled: Y / N Duplexed: Y / N

SOPS REQUESTED

Only signed and approved SOPs will be distributed*

Click to Select:

<input type="checkbox"/>	SOP Number	SOP Title	Method	Rev#	Revision Date	Next Rev
WET-CHEMISTRY						
<input checked="" type="checkbox"/>	NC-WC-0031	Cyanide, Automated Pyridine-Barbituric Acid <i>(analysis - also select NC-WC-0032)</i>	SW9012A EPA335.1, 335.2 SM4500CN-I CLP ILM03.0	4	02/11/99	02/00
<input checked="" type="checkbox"/>	NC-WC-0032	Cyanide, Distillation <i>(prep - also select NC-WC-0031)</i>	SW9012A EPA335.1, 335.2 SM4500CN-I SM4500CN-E CLP ILM03.0	8	02/04/99	02/00
<input checked="" type="checkbox"/>	NC-WC-0084	Ion Chromatography, Determination of Inorganic anions by	EPA300.0	2	02/11/99	02/00
METALS						
<input checked="" type="checkbox"/>	CORP-MT-0001	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses - UPDATE III	6010B 200.7	2	12/15/97 Change form 3/31/98 included	3/99
<input checked="" type="checkbox"/>	CORP-MT-0005NC	Mercury in Aqueous Samples by Cold Vapor Atomic Absorption (Prep and Analysis)	SW846 7470A MCAWW 245.1	1.1	04/19/97	3/98
<input checked="" type="checkbox"/>	CORP-MT-0007NC	Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy (Prep and Analysis)	SW846 7471A MCAWW 245.5	1.1	04/17/97	3/98
INORGANIC-PREP						
<input checked="" type="checkbox"/>	NC-IP-0004	Acid Digestion of Sediment, Sludges, and Soils for Metal Analysis by GFAA, FLAA or ICP	SW3050A EPA 200 Series	0	11/30/95	10/96

SOP REQUEST FORM

Click to Select:

<input type="checkbox"/>	SOP Number	SOP Title	Method	Rev#	Revision Date	Next Rev
<input checked="" type="checkbox"/>	NC-IP-0003	ICP & FLAA Prep for Total Metals (Aqueous)	EPA200.7;3005A SW3010A	1	12/08/95	11/96
ORGANIC PREP						
<input checked="" type="checkbox"/>	CORP-OP-0001NC	Extraction and Cleanup of Organic Compounds from Waters and Soils - UPDATE III	SW846 3500-ser; 3600-ser, 8151A, and 600 - series	3.1	07/31/98	07/99
GC/MS						
<input checked="" type="checkbox"/>	CORP-MS-0001	GC/MS Analysis Based on Method 8270C and 625 - UPDATE III	8270C and 625	2	12/15/97	12/98
<input checked="" type="checkbox"/>	CORP-MS-0002	Determination of Volatile Organics by GC/MS - UPDATE III	8260B, 624, and 524.2	2	12/15/97	12/98
GC						
<input checked="" type="checkbox"/>	CORP-GC-0001	Gas Chromatographic Analysis - UPDATE III	SW846 8000B, 8021B, 8081A, 8082, 8151A	5	12/07/98	11/99
SAMPLE CONTROL						
<input checked="" type="checkbox"/>	NC-SC-0005	Sample Receiving	N/A	4	06/02/97	5/98
QUALITY ASSURANCE						
<input checked="" type="checkbox"/>	QA-003	Quanterra® Quality Control Program	N/A	1	08/19/97	7/98

*If requirements indicate method deviations, then client-specific SOPs must be written and approved. These requests should be clearly stated as attachments to this form.

Special Requirements: Please email the SOPs (may take you a couple of emails due to the size of some of the SOPs) to Stacey Aubuchon. Make a copy of the cover page (stamped uncontrolled). The cover page along with some other information will be mailed to her tomorrow.

Please allow sufficient time for approval and processing.

QA DEPARTMENT USE ONLY:

Technical Contact(s): Opal Davis-Johnson

Request Completed By: Brenda R. Pettay-Kravetz Date 03/10/99

Cover Letter Prepared and Attached? Y N QA Director Approval: NA

1.

TABLE OF CONTENTS

1. SCOPE AND APPLICATION.....	4
2. SUMMARY OF METHOD	4
3. DEFINITIONS	5
4. INTERFERENCES	7
5. SAFETY	7
6. EQUIPMENT AND SUPPLIES	8
7. REAGENTS AND STANDARDS	11
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	12
9. QUALITY CONTROL	13
10. CALIBRATION AND STANDARDIZATION	17
11. PROCEDURE	20
12. DATA ANALYSIS AND CALCULATIONS	25
13. METHOD PERFORMANCE.....	31
14. POLLUTION PREVENTION.....	31
15. WASTE MANAGEMENT.....	32
16. REFERENCES.....	32
17. MISCELLANEOUS.....	32

APPENDIX A

APPLICATION OF METHOD 8260A TO THE DRINKING WATER ANALYTE LIST49

LIST OF TABLES

Table 1	Quanterra Primary Standard and Reporting Limits
Table 2	Quanterra Primary Standard Calibration Levels
Table 3	Quanterra Appendix IX Standard and Reporting Limits
Table 4	Quanterra Appendix IX Standard Calibration Levels
Table 5	Reportable Analytes for Quanterra Standard Tests, Primary Standard
Table 6	Reportable Analytes for Quanterra Standard Tests, Appendix IX Standard
Table 7	Internal Standards
Table 8	Surrogate Standards
Table 9	Matrix Spike / LCS Standards
Table 10	BFB Tune Criteria
Table 11	SPCC Compounds and Minimum Response Factors
Table 12	CCC Compounds
Table 13	Characteristic Ions
Table 14	Quanterra 8260A Drinking Water List Standard and Reporting Limits
Table 15	Internal Standards, Method 8260A Drinking Water List
Table 16	Surrogate Standards, Method 8260A Drinking Water List
Table 17	Calibration Levels, Method 8260A Drinking Water List

SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewaters, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.
- 1.2. This SOP is applicable to methods 8240B (capillary column) and 8260A.
- 1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.
- 1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. Directions are provided for analysis based on methods 8240B and 8260A. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 60 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 630 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1, 3 and 14.
- 1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

- 2.1. The differences between method 8240B and 8260A as performed at Quanterra are summarized here.
 - 2.1.1. Method 8240 was written as a packed column method; however, Quanterra has modified the method to use capillary columns for improved performance. Accordingly, when the surrogates and internal standards listed in this SOP are used, methods 8240B and 8260A are similar except for the minimum response factors listed in Table 11. **Both** methods may use 5 or 25 mL purge volumes, depending on the detection limits required, and both methods have the same calibration criteria.
 - 2.1.2. When a method 8260A analysis is requested, a target compound list based on that contained in method 524.2 is frequently requested. One of the target compounds, bromochloromethane, is a method 8240B internal standard, so alternative surrogates and internal standards must be used. Quanterra's standard analyte list, surrogates and internal standards for method 8260A (drinking water analyte list) are listed in Appendix A, Tables 14, 15, and 16.

- 2.2. The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer which is used to provide both qualitative and quantitative information.
- 2.3. If the above sample introduction techniques are not applicable, 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 in a purging chamber. It is then analyzed by purge and trap.
- 2.4. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature (40°C for soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.
- 2.5. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. If no changes to instrumental parameters are made, a batch may extend for a maximum of 24 hours. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

- 3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

A method blank consisting of all reagents added to the samples must be analyzed with each batch of 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.

3.3. 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. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

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. Each 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.

3.5. 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 aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

- 4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.
- 4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.
- 4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.
- 4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.
- 4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

- 5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.
- 5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Quanterra Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

- 5.3. Consult the Quanterra Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan) and a laboratory coat must be worn in the lab. 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. Disposable gloves shall not be reused.
- 5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:
 - 5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.
 - 5.4.2. Chemicals known to be flammable are: Methanol.
- 5.5. 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. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.
- 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.
- 5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Quanterra Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

- 6.1. Microsyringes: 10 μ L and larger, 0.006 inch ID needle.
- 6.2. Syringe: 5 mL glass with luerlok tip, if applicable to the purging device.

- 6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g
- 6.4. Glassware:
 - 6.4.1. Vials: 20 mL with screw caps and Teflon liners.
 - 6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.
- 6.5. Spatula: Stainless steel.
- 6.6. Disposable pipets: Pasteur.
- 6.7. pH paper: Wide range.
- 6.8. Gases:
 - 6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.
 - 6.8.2. Compressed air: Used for instrument pneumatics.
 - 6.8.3. Liquid nitrogen: Used for cryogenic cooling if necessary.
- 6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.
 - 6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated.
 - 6.9.2. Trap: The trap should be at least 25 cm long and have an inside diameter of at least 0.105 inch. Starting from the inlet, the trap should contain the following amounts of absorbents: 1/3 of 2,6-diphenylene oxide polymer (Tenax-GC, 60/80 mesh or equivalent), 1/3 of silica gel (Davison Chemical, 35-60 mesh, grade 15, or equivalent), and 1/3 coconut charcoal. It is recommended to use a trap that also 1.0 cm methyl silicone packing at the inlet to extend its life. If it is not necessary to analyze for any fluorocarbons then the charcoal phase can be replaced with the polymer. Other traps, such as Supelco's Vocarb 3000 and 4000, may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater or heated bath capable of maintaining the purge device at 40°C is used for low level soil analysis only.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming. The system must include or be interfaced to a purge and trap device. All GC carrier gas lines must be made from stainless steel or copper tubing.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53 ID Rtx-624 with 3 µm film thickness.

6.10.2.2. Column 2: 75 m x 0.53 ID DB-624 widebore with 3 µm film thickness.

6.10.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.4. GC/MS interface: In general glass jet separators are used but any interface that achieves all acceptance criteria may be used.

6.10.5. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.6. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the subambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C . Stock standards for gases must be replaced at least every week. Other stock standards must be replaced at least every 6 months.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored with minimal headspace and monitored for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. The standards are stored for a maximum of one week.

Note: By definition in this SOP, a stock standard is one that is stored in the freezer and is only opened in order to prepare working standards. A working standard is opened more frequently in order to prepare the calibration, spiking or tuning standards used at the instrument. Stock and working standards may be at the same concentration.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date. Once the ampoule is opened the expiration dates in Section 7.2.1.1 become effective.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

- 7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.
- 7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.
- 7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.
- 7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 25 ng/ μ L of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Standard 40 mL glass screw-cap VOA vials with Teflon-faced silicone septa may be used for both liquid and solid matrices. Solid samples may also be collected in wide-mouth glass jars with Teflon-lined caps. Samples should be introduced into the containers with minimum agitation to avoid loss of volatile compounds. For liquid samples, each VOA vial should be filled without introduction of bubbles. Fill until there is a meniscus over the lip of the vial. The lid with septum (Teflon side toward the sample) should be tightened onto the vial. After tightening the lid, the vial should be inverted and tapped to check for air bubbles. If there are any air bubbles present the sample must be retaken. Sample containers for solid samples should be filled as completely as possible with minimum air space.
- 8.2. Water samples are preserved with HCl. Aromatic compounds are particularly susceptible to biodegradation at normal pH. The pH of the sample should be adjusted to less than 2 with HCl in the field at the time of sampling.
 - 8.2.1. If the sample is unpreserved every reasonable effort should be made to analyze the sample within 7 days from sampling. The condition should be documented as an anomaly and the normal holding time applied. If the sample requires analysis of aromatic compounds the potential impact on the data must be documented.
- 8.3. All samples must be iced or refrigerated at $4^{\circ} \pm 2^{\circ}\text{C}$ from the time of collection until analysis or extraction.
- 8.4. For shipping information, see the facility Sample Procurement Protocol SOP.
- 8.5. The holding time is fourteen days from sampling to the completion of analysis.

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. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery ± 3 standard deviations for surrogates and LCS, and mean recovery ± 2 standard deviations for matrix spikes. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference ± 2 standard deviations.

- 9.2.1. For medium level soils only, these limits do not apply to dilutions. Surrogate and matrix spike recoveries for medium level soils will be reported unless the dilution is more than 5X.
- 9.2.2. 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.
- 9.2.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 16. 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 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.

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.

- 9.3.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and reparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.
- 9.3.2. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks

For each batch of samples, analyze a method blank. The method blank is normally analyzed immediately after the calibration standards. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 9.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 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) 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.
- 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 done in consultation with the client.

- 9.4.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.
- 9.4.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.
- 9.4.3. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS is normally analyzed immediately after the method blank. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. 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 re-preparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)
 - If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.
- 9.5.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective action.
 - 9.5.2. 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.

9.6. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the matrix spike/spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. 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. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.2. Recommended Instrument Conditions

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 1 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C
-------------	-------

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C

Final Hold Time: 2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. The calibration levels for a 5 mL purge are: 10, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Calibration levels for a 25 mL purge are 2, 5, 10, 30, and 60 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 4, and 17 list the calibration levels for each analyte.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6).

10.4.3. Internal standard calibration is used. The internal standards are listed in Tables 7 and 15. Target compounds should reference the nearest internal standard. In particular, the SPCC compounds bromoform and 1,1,2,2-tetrachloroethane reference chlorobenzene-d5, not 1,4-difluorobenzene, which was appropriate for packed column analysis. Note different internal standards are used for the method 8260A drinking water analyte list (Appendix A). Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 30% criterion.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. If the %RSD of $\geq 80\%$ of the analytes in the calibration is $\leq 15\%$, then all analytes may use average response factor for calibration.

10.4.6.1. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD $> 15\%$ for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve (e.g. $R^2 > 0.995$) then the appropriate curve should be used for quantitation.

10.4.6.2. If less than 80% of the analytes in the calibration have %RSD $\leq 15\%$, then calibration on a curve must be used for all analytes with %RSD $> 15\%$. The analyst should consider instrument maintenance to improve the linearity of response.

10.4.7. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. A separate five point calibration must be prepared for analysis of low level soils. Each standard is prepared as in Section 10.4.1, except that the standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated) calibration curve.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

- 10.5.3. The % drift of the CCCs must be < 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all analytes must be \leq 50% with allowance for up to six target analytes to have % drift > 50%.
- 10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 20% criterion.
- 10.5.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).
- 10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4. the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.
- 10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

- 11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- 11.1.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

- 11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.
- 11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a 5 or 25 mL Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the 5/25 mL syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the 5/25 mL mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 5 μ L of sample then serial dilutions must be made in volumetric flasks.
- 11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

- 11.3.1. All analysis conditions for samples must be the same as for the calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).
- 11.3.2. Sample Preparation Procedure
- 11.3.2.1. Samples fall into three general categories: waters, low-level soils, and medium-level soils or wastes. For waters and low-level soils, no sample preparation is necessary. For those soils which contain greater than 1 mg/kg of individual purgeable compounds, a medium-level preparation is necessary.
- 11.3.3. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.
- 11.3.3.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.
- 11.3.3.2. One MS/MSD pair does not count towards the maximum 20 samples in the batch. Additional client requested MS/MSD samples do count towards the maximum 20 samples.

- 11.3.3.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.3.4. Water Samples

- 11.3.4.1. All samples and standard solutions must be at ambient temperature before analysis.
- 11.3.4.2. Fill a 5 or 25 mL syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq 5 \mu\text{L}$. Check and document the pH of the remaining sample. *Record the volume to the nearest 0.1 mL*
- 11.3.4.3. Add 250 ng of each internal and surrogate standard (10 μL of a 25 $\mu\text{g}/\text{mL}$ solution, refer to Tables 7, 8, 15 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 $\mu\text{g}/\text{L}$ solution for a 5 mL sample, and a 10 $\mu\text{g}/\text{L}$ solution for a 25 mL sample). Inject the sample into the purging chamber.
- 11.3.4.3.1. For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 μL of the 25 $\mu\text{g}/\text{mL}$ TCLP spiking solution.
- 11.3.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).
- 11.3.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.
- 11.3.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.3.5. Medium-Level Soil/Sediment and Waste Samples

- 11.3.5.1. Sediments, soils and waste that are insoluble in methanol.
- 11.3.5.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 4 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.
- 11.3.5.1.2. Quickly add 9 mL of methanol, and 1 mL of surrogate spiking solution to bring the final volume of methanol to 10 mL. For an LCS or MS/MSD

sample add 8 mL of methanol, 1 mL of surrogate spike solution, and 1 mL of matrix spike solution. Cap the vial and vortex to mix thoroughly.

NOTE: Sections 11.3.5.1.1 and 11.3.5.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.3.5.2. Liquid wastes that are soluble in methanol and insoluble in water.

11.3.5.2.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.3.5.2.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.3.5.3. Fill a 5 mL syringe with 5 mL of reagent water. Add 100 μ L (or less if a dilution is required) of methanol extract from the sample preparation in Section 11.3.5.1 or 11.3.5.2. If less than 5 μ L of the methanol extract is required, then an intermediate dilution is required. Add 10 μ L of the 25 μ g/mL internal standard solution. (Note that the combined internal standard/surrogate standard solution is not used since surrogates have been added previously.) Inject the sample into the purging chamber and proceed with the analysis as per Sections 11.3.4.4 and 11.3.4.5.

11.3.6. Low-Level Soils

11.3.6.1. This is designed for samples containing individual purgeable compounds of < 2 mg/kg. It is limited to soil/sediment samples and waste that is of a similar consistency (granular and porous). Weigh 5 g of the sample into a tared purge vessel. If a dilution is required, a smaller sample amount can be analyzed, down to a minimum of 0.5 g. Any soil sample requiring further dilution must be run as a medium-level soil. Add 5 mL of reagent water to which 10 μ L of the 25 μ g/mL internal standard/surrogate standard solution has been added. *Record the weight to the nearest 0.1 g.* Proceed with the analysis as per Sections 11.3.4.4 and 11.3.4.5. Note: Up to 10 mL of reagent water may be added to a soil sample to increase purge gas interaction with the sample. The initial calibration curve and all standards, blanks, and samples using that ICAL must have the same amount of reagent water added. The amount of internal standards and surrogate standards added will not be changed.

11.4. Initial review and corrective actions

- 11.4.1. If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.
- 11.4.2. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.
- 11.4.3. Internal standard response in each sample should be within 50% to 200% of the response in the preceding continuing calibration standard.
 - 11.4.3.1. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis may not be necessary. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected. In any event, the reason for accepting the sample analysis must be documented. Some clients may require reanalysis of all samples with internal standard criteria outside the 50-200% criteria. Consideration should be given to reanalyzing at a dilution to reduce matrix effects. It is only necessary to reanalyze once to confirm matrix effect.
- 11.4.4. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.5. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the 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.

11.5.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the peaks in the level 3 standard, then the sample should be reanalyzed at a more concentrated dilution.

11.5.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within $\pm 30\%$ between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TIC's)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification of *all non-*

target peaks eluting within one minute after the elution time of the last target compound (excluding the internal and surrogate standards). Guidelines are:

- 12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
- 12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).
- 12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.
- 12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- 12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)
- 12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\frac{\sum_{i=1}^N (X_i - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = Mean of RF values in the curve

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\% \text{ Drift} = \frac{C_{\text{expected}} - C_{\text{found}}}{C_{\text{expected}}} \times 100$$

Where

C_{expected} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq 15\%$.

12.3.5.1. Calculation of concentration using Quadratic fit

Equation 5

$$\text{Concentration } \mu\text{g} / \text{L} = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\text{Concentration } \mu\text{g} / \text{L} = A + Bx$$

12.3.5.3. Calculation of concentration using Average Response Factors

Equation 7

$$\text{Concentration } \mu\text{g} / \text{L} = \frac{x}{RF}$$

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_s)(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_s = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, ml.

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_s)(I_s)(V_t)(1000)(D)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_s , I_s , D , A_{is} , same as for water.

V_t = Volume of total extract, mL (Typically 10 mL)

V_a = Volume of extract added for purging, μ L

W_s = Weight of sample extracted, g

$$D = \frac{100 - \% \text{moisture}}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_s)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_s , I_s , A_{is} , same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\text{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

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 soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

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. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be $\leq 15\%$ for each analyte, and the % recovery should be within 80-120%.

13.2.3. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of method 8240B, paragraph 8.3.5 of method 8260A). If the recovery or precision is outside the limits in the reference methods, 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

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 according to the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

- 16.1. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8240B, Update II, September 1994.
- 16.2. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994.

17. MISCELLANEOUS

17.1. Modifications from the reference method

- 17.1.1. Method 8240B has been modified in this SOP to use capillary columns rather than the packed columns listed in the reference method. This provides improved separation of the target analytes and improved detection limits.
- 17.1.2. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.
- 17.1.3. The internal standard control criteria of 50% to 200% is applied to each sample rather than the subsequent continuing calibration standard as recommended in the reference method.
- 17.1.4. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.
- 17.1.5. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
- 17.1.6. Method 8260A recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

17.1.7. Matrix spikes and surrogates are spiked at the levels specified in methods 3500 and 5030.

17.1.8. SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In addition most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, this SOP allows the use of average response factors of 80% if the analytes have %RSDs \leq 15%. Modifications from previous revision

17.1.9. If at least 80% of the analytes in the initial calibration have %RSD < 15%, average response factor calibration may be used for all analytes.

17.2. 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.

17.3.

Flow diagrams

17.3.1. Initial Demonstration and MDL

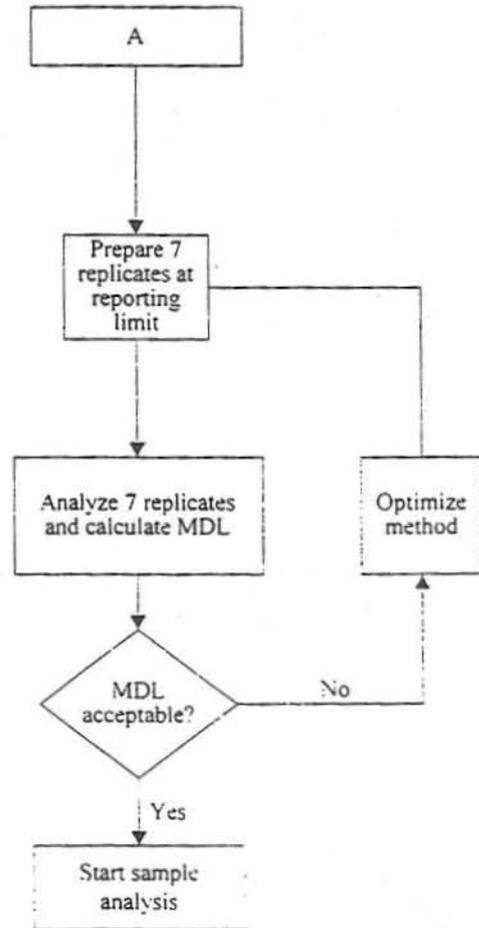
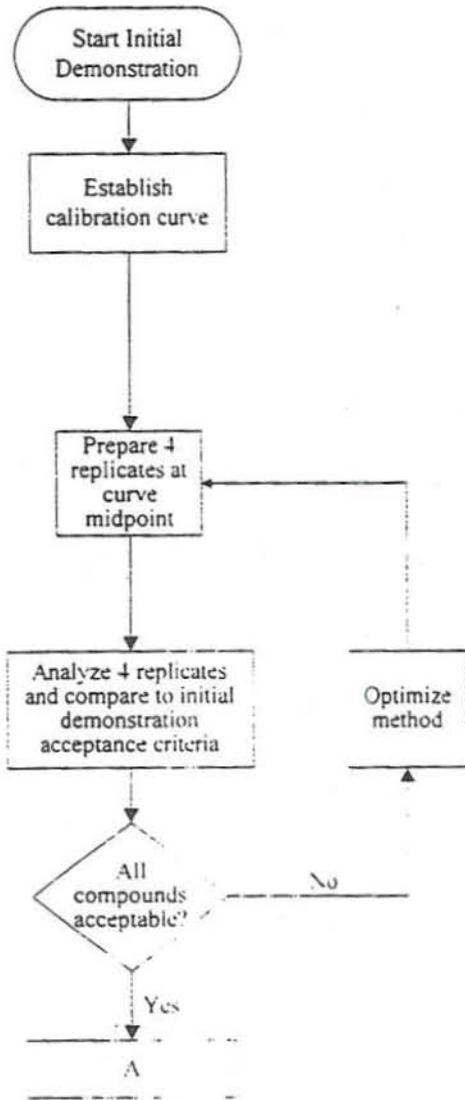


Table 1
Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits ¹			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	1200
Chloromethane	74-87-3	10	2	10	1200
Bromomethane	74-83-9	10	2	10	1200
Vinyl chloride	75-01-4	10	2	10	1200
Chloroethane	75-00-3	10	2	10	1200
Trichlorofluoromethane	75-69-4	10	2	10	1200
Acrolein	107-02-8	100	20	100	12000
Acetone	67-64-1	20	10	20	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	620
Ethanol	64-17-5	500	200	500	62,000
Iodomethane	74-88-4	5	1	5	620
Carbon disulfide	75-15-0	5	1	5	620
Methylene chloride	75-09-2	5	1	5	620
tert-Butyl alcohol	75-65-0	200	50	200	25,000
1,1-Dichloroethene	75-35-4	5	1	5	620
1,1-Dichloroethane	75-34-3	5	1	5	620
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	310
Acrylonitrile	107-13-1	100	20	100	12000
Methyl tert-butyl ether (MTBE)	1634-04-4	20	5	20	2500
Hexane	110-54-3	5	1	5	620
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	620
Tetrahydrofuran	109-99-9	20	5	20	2500
Chloroform	67-66-3	5	1	5	620
1,2-Dichloroethane	107-06-2	5	1	5	620
Dibromomethane	74-95-3	5	1	5	620
2-Butanone	78-93-3	20	5	20	2500
1,4-Dioxane	123-91-1	500	200	500	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	620
Carbon tetrachloride	56-23-5	5	1	5	620
Bromodichloromethane	75-27-4	5	1	5	620
1,2-Dichloropropane	78-87-5	5	1	5	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	620
Trichloroethene	79-01-6	5	1	5	620

Table 1
Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits ¹			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dibromochloromethane	124-48-1	5	1	5	620
1,2-Dibromoethane	106-93-4	5	1	5	620
1,2,3-Trichloropropane	96-18-4	5	1	5	620
1,1,2-Trichloroethane	79-00-5	5	1	5	620
Benzene	71-43-2	5	1	5	620
Ethylmethacrylate	97-63-2	5	1	5	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	620
Bromoform	75-25-2	5	1	5	620
4-Methyl-2-pentanone	108-10-1	20	5	20	2500
2-Hexanone	591-78-6	20	5	20	2500
Tetrachloroethene	127-18-4	5	1	5	620
Toluene	108-88-3	5	1	5	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	620
2-Chloroethyl vinyl ether	110-75-8	N/A ²	N/A	50	6200
Vinyl acetate	108-05-4	10	2	10	1200
Chlorobenzene	108-90-7	5	1	5	620
Ethylbenzene	100-41-4	5	1	5	620
Styrene	100-42-5	5	1	5	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	620
m and p Xylenes		2.5	0.5	2.5	310
o-xylene	95-47-6	2.5	0.5	2.5	310
Total xylenes	1330-20-7	5	1	5	620
1,3-Dichlorobenzene	541-73-1	5	1	5	620
1,4-Dichlorobenzene	106-46-7	5	1	5	620
1,2-Dichlorobenzene	95-50-1	5	1	5	620

Reporting limits listed for soil sediment are based on wet weight. The reporting limits calculated by the laboratory for soil sediment, calculated on dry weight basis, will be higher.

² 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2
Quanterra Primary Standard Calibration Levels, 5 mL purge¹

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	10	20	50	100	200
Toluene-d8 (Surrogate)	10	20	50	100	200
4-Bromofluorobenzene (Surrogate)	10	20	50	100	200
Dichlorodifluoromethane	10	20	50	100	200
Chloromethane	10	20	50	100	200
Bromomethane	10	20	50	100	200
Vinyl chloride	10	20	50	100	200
Chloroethane	10	20	50	100	200
Trichlorofluoromethane	10	20	50	100	200
Acrolein	100	200	500	1000	2000
Acetone	10	20	50	100	200
Trichlorotrifluoroethane	10	20	50	100	200
Ethanol	500	1000	5000	10,000	20,000
Iodomethane	10	20	50	100	200
Carbon disulfide	10	20	50	100	200
Methylene chloride	10	20	50	100	200
tert-Butyl alcohol	200	400	1,000	2,000	4,000
1,1-Dichloroethene	10	20	50	100	200
1,1-Dichloroethane	10	20	50	100	200
trans-1,2-Dichloroethene	10	20	50	100	200
Acrylonitrile	100	200	500	1,000	2,000
Methyl tert-butyl ether (MTBE)	10	20	50	100	200
Hexane	10	20	50	100	200
cis-1,2-Dichloroethene	10	20	50	100	200
Tetrahydrofuran	10	20	50	100	200
Chloroform	10	20	50	100	200
1,2-Dichloroethane	10	20	50	100	200
Dibromomethane	10	20	50	100	200
2-Butanone	10	20	50	100	200
1,4-Dioxane	500	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	10	20	50	100	200
Carbon tetrachloride	10	20	50	100	200
Bromodichloromethane	10	20	50	100	200
1,2-Dichloropropane	10	20	50	100	200
cis-1,3-Dichloropropene	10	20	50	100	200

Table 2
Quanterra Primary Standard Calibration Levels, 5 mL purge¹

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Trichloroethene	10	20	50	100	200
Dibromochloromethane	10	20	50	100	200
1,2-Dibromoethane	10	20	50	100	200
1,2,3-Trichloropropane	10	20	50	100	200
1,1,2-Trichloroethane	10	20	50	100	200
Benzene	10	20	50	100	200
Ethylmethacrylate	10	20	50	100	200
trans-1,3-Dichloropropene	10	20	50	100	200
Bromoform	10	20	50	100	200
4-Methyl-2-pentanone	10	20	50	100	200
2-Hexanone	10	20	50	100	200
Tetrachloroethene	10	20	50	100	200
Toluene	10	20	50	100	200
1,1,2,2-Tetrachloroethane	10	20	50	100	200
2-Chloroethyl vinyl ether	20	40	100	200	400
Vinyl acetate	10	20	50	100	200
Chlorobenzene	10	20	50	100	200
Ethylbenzene	10	20	50	100	200
Styrene	10	20	50	100	200
t-1,4-Dichloro-2-butene	10	20	50	100	200
m and p Xylenes	20	40	100	200	400
o-xylene	10	20	50	100	200
1,3-Dichlorobenzene	10	20	50	100	200
1,4-Dichlorobenzene	10	20	50	100	200
1,2-Dichlorobenzene	10	20	50	100	200

¹ Levels for 25 mL purge are 5 times lower in all cases

Table 3

Quanterra Appendix IX Standard and Reporting Limits, 5 mL purge¹

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25 mL water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	1,200
Acetonitrile	75-05-8	100	20	100	12,000
Dichlorofluoromethane		10	2	10	1,200
Isopropyl ether	108-20-3	50	10	50	6,200
Chloroprene	126-99-8	5	1	5	620
n-Butanol	71-36-3	200	50	200	25,000
Propionitrile	107-12-0	20	4	20	2,500
Methacrylonitrile	126-98-7	5	1	5	620
Isobutanol	78-83-1	200	50	200	25,000
Methyl methacrylate	80-62-6	5	1	5	620
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	620
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	1,200
Ethyl ether	60-29-7	10	2	10	1,200
Ethyl Acetate	141-78-6	20	4	20	2,500
2-Nitropropane	79-46-9	10	2	10	1,200
Cyclohexanone	108-94-1	N/A ²	N/A ²	N/A ²	N/A ²
Isopropylbenzene	98-82-8	5	1	5	620

¹ Levels for 25 mL purge are 5 times lower in all cases

² Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4
Quanterra Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	10	20	50	100	200
Acetonitrile	100	200	500	1,000	2,000
Dichlorofluoromethane	10	20	50	100	200
Isopropyl ether	50	100	250	500	1,000
Chloroprene	10	20	50	100	200
n-Butanol	200	400	1,000	2,000	4,000
Propionitrile	20	40	100	200	400
Methacrylonitrile	10	20	50	100	200
Isobutanol	200	400	1,000	2,000	4,000
Methyl methacrylate	10	20	50	100	200
1,1,1,2-Tetrachloroethane	10	20	50	100	200
1,2-Dibromo-3-chloropropane	20	40	100	200	400
Ethyl ether	10	20	50	100	200
Ethyl Acetate	20	40	100	200	400
2-Nitropropane	20	40	100	200	400
Cyclohexanone	100	200	500	1,000	2,000
Isopropylbenzene	10	20	50	100	200

Table 5
Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butyl alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1					X
Methyl tert-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-	X		X	X	X

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
	5					
Trichloroethene	79-01-6	X	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,1,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for Quanterra Standard Tests, Appendix IX standard

Compound	Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7
Internal Standards

	Standard Concentration $\mu\text{g/mL}$	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Bromochloromethane	25	128	128
1,4-Difluorobenzene	25	114	114
Chlorobenzene d5	25	117	119

Notes:

- 1) 10 μL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50 $\mu\text{g/L}$ for a 5 mL purge or 10 $\mu\text{g/L}$ for a 25 mL purge.
- 2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8
Surrogate Standards

Surrogate Compounds	Standard Concentration $\mu\text{g/mL}$
1,2-Dichloroethane-d ₂	25
Toluene-d ₈	25
4-Bromofluorobenzene	25

Notes:

- 1) 10 μL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50 $\mu\text{g/L}$ for a 5 mL purge or 10 $\mu\text{g/L}$ for a 25 mL purge.
- 2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.
- 3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9

Matrix Spike / LCS Compounds

Compound	Standard Concentration $\mu\text{g/mL}$
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

- 1) 10 μL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50 $\mu\text{g/L}$ for a 5 mL purge or 10 $\mu\text{g/L}$ for a 25 mL purge.
- 2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA department.

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Note: The Target software in use in the laboratory lists the acceptance criteria for mass 176 as "95.00 - 101.00 of mass 174". In the event that the 174-176 ratio rounds to 101.00%, the BFB may not have met the method criteria, and may not be flagged as failing on the Target report.

Table 11
SPCC Compounds and Minimum Response Factors

Compound	8240B Min. RF	8260A Min. RF
Chloromethane	0.300	0.100
1,1-Dichloroethane	0.300	0.100
Bromoform	>0.100	>0.100
1,1,2,2-Tetrachloroethane	0.300	0.300
Chlorobenzene	0.300	0.300

Table 12
CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0

Table 13
Characteristic ions

Compound	Primary*	Secondary	Tertiary
Bromochloromethane (Internal Standard)	128	49	130, 51
1,2-Dichloroethane-d ₁ (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101, 103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58

Table 13
Characteristic ions

Compound	Primary*	Secondary	Tertiary
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl tert butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
1,4-Difluorobenzene (Internal Standard)	114	63	88
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Chlorobenzene-d ₄ (Internal Standard)	117, 119	117, 119	
Toluene-d ₈ (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65

Table 13
Characteristic ions

Compound	Primary*	Secondary	Tertiary
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification

Table 14
Quanterra Primary Standard Calibration Levels

25 mL Purge

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Dichlorodifluoromethane	2	4	10	20	40
Chloromethane	2	4	10	20	40
Bromomethane	2	4	10	20	40
Vinyl chloride	2	4	10	20	40
Chloroethane	2	4	10	20	40
Trichlorofluoromethane	2	4	10	20	40
Acrolein	20	40	100	200	400
Acetone	20	40	100	200	400
Trichlorofluoroethane	2	4	10	20	40
Ethanol	100	200	500	1000	2000
Iodomethane	2	4	10	20	40
Carbon disulfide	2	4	10	20	40
Methylene chloride	2	4	10	20	40
tert-Butyl alcohol	40	80	200	400	800
1,1-Dichloroethene	2	4	10	20	40
1,1-Dichloroethane	2	4	10	20	40
trans-1,2-Dichloroethene	2	4	10	20	40
Acrylonitrile	20	40	100	200	400
Methyl tert-butyl ether (MTBE)	2	4	10	20	40
Hexane	2	4	10	20	40

Table 14
Quanterra Primary Standard Calibration Levels

25 mL Purge

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
cis-1,2-Dichloroethene	2	4	10	20	40
Tetrahydrofuran	2	4	10	20	40
Chloroform	2	4	10	20	40
1,2-Dichloroethane	2	4	10	20	40
Dibromomethane	2	4	10	20	40
2-Butanone	20	40	100	200	400
1,4-Dioxane	100	200	500	1000	2000
1,1,1-Trichloroethane	2	4	10	20	40
Carbon tetrachloride	2	4	10	20	40
Bromodichloromethane	2	4	10	20	40
1,2-Dichloropropane	2	4	10	20	40
cis-1,3-Dichloropropene	2	4	10	20	40
Trichloroethene	2	4	10	20	40
Dibromochloromethane	2	4	10	20	40
1,2-Dibromoethane	2	4	10	20	40
1,2,3-Trichloropropane	2	4	10	20	40
1,1,2-Trichloroethane	2	4	10	20	40
Benzene	2	4	10	20	40
Ethylmethacrylate	2	4	10	20	40
trans-1,3-Dichloropropene	2	4	10	20	40
Bromoform	2	4	10	20	40

Table 14
Quanterra Primary Standard Calibration Levels

25 mL Purge

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
4-Methyl-2-pentanone	20	40	100	200	400
2-Hexanone	20	40	100	200	400
Tetrachloroethene	2	4	10	20	40
Toluene	2	4	10	20	40
1,1,2,2-Tetrachloroethane	2	4	10	20	40
2-Chloroethyl vinyl ether	4	8	20	40	80
Vinyl acetate	2	4	10	20	40
Chlorobenzene	2	4	10	20	40
Ethylbenzene	2	4	10	20	40
Styrene	2	4	10	20	40
trans-1,4-Dichloro-2-butene	2	4	10	20	40
m and p Xylenes	4	8	20	40	80
o-Xylene	2	4	10	20	40
1,3-Dichlorobenzene	2	4	10	20	40
1,4-Dichlorobenzene	2	4	10	20	40
1,2-Dichlorobenzene	2	4	10	20	40
Acetonitrile	20	40	100	200	400
Bromochloromethane	2	4	10	20	40

1. SUMMARY

A target analyte list based on the list in method 524.2 is frequently requested for analysis by method 8260A. Quanterra's standard analyte list for this test, and the internal and surrogate standards used, are listed in Tables below. In all other respects the method is as described in the main body of this SOP. Note that this SOP is *not* appropriate for drinking water analysis by method 524.2.

Table 14
Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits ¹			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	1200
Chloromethane	74-87-3	10	2	10	1200
Bromomethane	74-83-9	10	2	10	1200
Vinyl chloride	75-01-4	10	2	10	1200
Chloroethane	75-00-3	10	2	10	1200
Trichlorofluoromethane	75-69-4	10	2	10	1200
Acetone ¹	67-64-1	20	10	20	2500
Methylene chloride	75-09-2	5	2	5	620
1,1-Dichloroethene	75-35-4	5	1	5	620
1,1-Dichloroethane	75-34-3	5	1	5	620
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	310
Methyl <i>tert</i> -butyl ether (MTBE)	1634-04-4	20	5	20	620
2,2-Dichloropropane	590-20-7	5	1	5	620
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	620
Chloroform	67-66-3	5	1	5	620
Bromochloromethane	74-97-5	5	1	5	620
1,2-Dichloroethane	107-06-2	5	1	5	620
Dibromomethane	74-95-3	5	1	5	620
2-Butanone ¹	78-93-3	20	5	20	2500
1,1,1-Trichloroethane	71-55-6	5	1	5	620
Carbon tetrachloride	56-23-5	5	1	5	620
Bromodichloromethane	75-27-4	5	1	5	620
1,2-Dichloropropane	78-87-5	5	1	5	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	620

Table 14
Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits ¹			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Trichloroethene	79-01-6	5	1	5	620
Dibromochloromethane	124-48-1	5	1	5	620
1,2-Dibromoethane	106-93-4	5	1	5	620
1,2,3-Trichloropropane	96-18-4	5	1	5	620
1,1,2-Trichloroethane	79-00-5	5	1	5	620
Benzene	71-43-2	5	1	5	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	620
Bromoform	75-25-2	5	1	5	620
4-Methyl-2-pentanone ¹	108-10-1	20	5	20	2500
2-Hexanone ¹	591-78-6	20	5	20	2500
Tetrachloroethene	127-18-4	5	1	5	620
Toluene	108-88-3	5	1	5	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	620
Chlorobenzene	108-90-7	5	1	5	620
Ethylbenzene	100-41-4	5	1	5	620
Styrene	100-42-5	5	1	5	620
m and p Xylenes		2.5	0.5	2.5	310
o-xylene	95-47-6	2.5	0.5	2.5	310
Total xylenes	1330-20-7	5	1	5	620
Isopropylbenzene	98-82-8	5	1	5	620
Bromobenzene	108-86-1	5	1	5	620
n-Propylbenzene	103-65-1	5	1	5	620
2-Chlorotoluene	95-49-8	5	1	5	620
4-Chlorotoluene	106-43-4	5	1	5	620
1,3,5-Trimethylbenzene	108-67-8	5	1	5	620
tert-Butylbenzene	98-06-6	5	1	5	620
1,2,4-Trimethylbenzene	95-63-6	5	1	5	620
sec-butylbenzene	135-98-8	5	1	5	620
1,3-Dichlorobenzene	541-73-1	5	1	5	620
1,4-Dichlorobenzene	106-46-7	5	1	5	620
1,2-Dichlorobenzene	95-50-1	5	1	5	620
4-Isopropyltoluene	99-87-6	5	1	5	620
n-Butylbenzene	104-51-8	5	1	5	620
1,2-Dibromo-3-chloropropane	96-12-8	5	1	5	620
1,2,4-Trichlorobenzene	120-82-1	5	1	5	620

Table 14

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits ¹			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Napthalene	91-20-3	5	1	5	620
Hexachlorobutadiene	87-68-3	5	1	5	620
1,2,3-Trichlorobenzene	87-61-6	5	1	5	620

¹ Not included on the method 524.2 analyte list, but includes in the calibration standard as an add on frequently requested by method 8260A.

Table 15

Internal Standards, Method 8260A Drinking water list

	Standard Concentration µg/mL	Quantitation ion
Fluorobenzene	25	128
Chlorobenzene-d5	25	114
1,4-Dichlorobenzene-d4	25	119

Notes:

- 1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg L for a 5 mL purge or 10 µg/L for a 25 mL purge.
- 2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 16
Surrogate Standards, Method 8260B Drinking water list

Surrogate Compounds	Standard Concentration $\mu\text{g/mL}$
1,2-Dichloroethane- d_2	25
Toluene- d_8	25
4-Bromofluorobenzene	25

Notes:

- 1) 10 μL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50 $\mu\text{g/L}$ for a 5 mL purge or 10 $\mu\text{g/L}$ for a 25 mL purge.
- 2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 17
Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
	5 mL	25 mL								
Dichlorodifluoromethane	20	4	40	10	100	20	200	60	400	120
Chloromethane	20	4	40	10	100	20	200	60	400	120
Bromomethane	20	4	40	10	100	20	200	60	400	120
Vinyl chloride	20	4	40	10	100	20	200	60	400	120
Chloroethane	20	4	40	10	100	20	200	60	400	120
Trichlorofluoromethane	20	4	40	10	100	20	200	60	400	120
Acetone ¹	20	4	40	10	100	20	200	60	400	120
Methylene chloride	10	2	20	5	50	10	100	30	200	60
1,1-Dichloroethene	10	2	20	5	50	10	100	30	200	60
1,1-Dichloroethane	10	2	20	5	50	10	100	30	200	60
trans-1,2-Dichloroethene	10	2	20	5	50	10	100	30	200	60
Methyl <i>tert</i> -butyl ether (MTBE)	20	4	40	10	100	20	200	60	400	120
2,2-Dichloropropane	10	2	20	5	50	10	100	30	200	60
cis-1,2-Dichloroethene	10	2	20	5	50	10	100	30	200	60

Table 17

Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
Chloroform	10	2	20	5	50	10	100	30	200	60
Bromochloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dichloroethane	10	2	20	5	50	10	100	30	200	60
Dibromomethane	10	2	20	5	50	10	100	30	200	60
2-Butanone ¹	20	4	40	10	100	20	200	60	400	120
1,1,1-Trichloroethane	10	2	20	5	50	10	100	30	200	60
Carbon tetrachloride	10	2	20	5	50	10	100	30	200	60
Bromodichloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dichloropropane	10	2	20	5	50	10	100	30	200	60
cis-1,3-Dichloropropene	10	2	20	5	50	10	100	30	200	60
Trichloroethene	10	2	20	5	50	10	100	30	200	60
Dibromochloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dibromoethane	10	2	20	5	50	10	100	30	200	60
1,2,3-Trichloropropane	10	2	20	5	50	10	100	30	200	60
1,1,2-Trichloroethane	10	2	20	5	50	10	100	30	200	60
Benzene	10	2	20	5	50	10	100	30	200	60
trans-1,3-Dichloropropene	10	2	20	5	50	10	100	30	200	60
Bromoform	10	2	20	5	50	10	100	30	200	60
4-Methyl-2-pentanone ¹	20	4	40	10	100	20	200	60	400	120
2-Hexanone ¹	20	4	40	10	100	20	200	60	400	120
Tetrachloroethene	10	2	20	5	50	10	100	30	200	60
Toluene	10	2	20	5	50	10	100	30	200	60
1,1,2,2-Tetrachloroethane	10	2	20	5	50	10	100	30	200	60
Chlorobenzene	10	2	20	5	50	10	100	30	200	60
Ethylbenzene	10	2	20	5	50	10	100	30	200	60
Styrene	10	2	20	5	50	10	100	30	200	60
m and p Xylenes	10	2	20	5	50	10	100	30	200	60
o-xylene	10	2	20	5	50	10	100	30	200	60

Table 17
Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Isopropylbenzene	10 2	20 5	50 10	100 30	200 60
Bromobenzene	10 2	20 5	50 10	100 30	200 60
n-Propylbenzene	10 2	20 5	50 10	100 30	200 60
2-Chlorotoluene	10 2	20 5	50 10	100 30	200 60
4-Chlorotoluene	10 2	20 5	50 10	100 30	200 60
1,3,5-Trimethylbenzene	10 2	20 5	50 10	100 30	200 60
tert-Butylbenzene	10 2	20 5	50 10	100 30	200 60
1,2,4-Trimethylbenzene	10 2	20 5	50 10	100 30	200 60
sec-butylbenzene	10 2	20 5	50 10	100 30	200 60
1,3-Dichlorobenzene	10 2	20 5	50 10	100 30	200 60
1,4-Dichlorobenzene	10 2	20 5	50 10	100 30	200 60
1,2-Dichlorobenzene	10 2	20 5	50 10	100 30	200 60
4-Isopropyltoluene	10 2	20 5	50 10	100 30	200 60
n-Butylbenzene	10 2	20 5	50 10	100 30	200 60
1,2-Dibromo-3-chloropropane	10 2	20 5	50 10	100 30	200 60
1,2,4-Trichlorobenzene	10 2	20 5	50 10	100 30	200 60
Napthalene	10 2	20 5	50 10	100 30	200 60
Hexachlorobutadiene	10 2	20 5	50 10	100 30	200 60
1,2,3-Trichlorobenzene	10 2	20 5	50 10	100 30	200 60

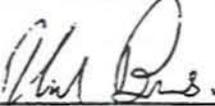
Not included in the Quanterra Standard test, but included in the standard as a frequently requested add-on.

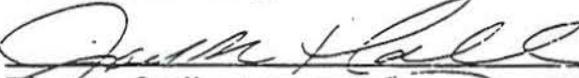
QUANTERRA STANDARD OPERATING PROCEDURE

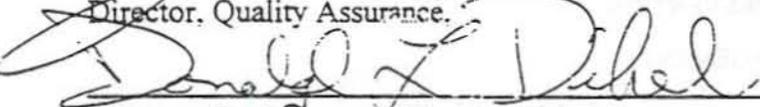
**TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY,
SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES,
SW-846 METHOD 6010B AND EPA METHOD 200.7**

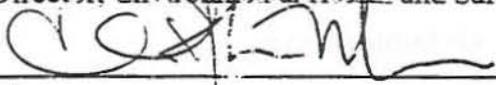
(SUPERSEDES: REVISION 1)

Prepared by: Sevda Aleckson and Bill Nasir

Reviewed by: 
Director, Technology

Approved by: 
Director, Quality Assurance.

Approved by: 
Director, Environmental Health and Safety,

Approved by: 
Management,

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

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	7
9. QUALITY CONTROL.....	8
10. CALIBRATION AND STANDARDIZATION	14
11. PROCEDURE	15
12. DATA ANALYSIS AND CALCULATIONS.....	7
13. METHOD PERFORMANCE.....	23
14. POLLUTION PREVENTION	23
15. WASTE MANAGEMENT.....	24
16. REFERENCES	24
17. MISCELLANEOUS (TABLES, APPENDICES, ETC.).....	24

LIST OF APPENDICES:

APPENDIX A - TABLES	30
APPENDIX B - QUANTERRA ICP DATA REVIEW CHECKLIST.....	40
APPENDIX C - TJA CALIBRATION RECOMMENDATION.....	42
APPENDIX D - MSA GUIDANCE	44
APPENDIX E - TROUBLESHOOTING GUIDE.....	46
APPENDIX F - CONTAMINATION CONTROL GUIDELINES	48
APPENDIX G - PREVENTATIVE MAINTENANCE.....	50

1. SCOPE AND APPLICATION

- 1.1. This procedure describes the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) using SW-846 protocol Method 6010A and Methods for Chemical Analysis of Waters and Wastes protocol, Method 200.7 as published in 40 CFR Part 136, Appendix C. Table I of Appendix A lists the elements approved for analysis by Methods 6010A and 200.7. Additional elements may be analyzed under Methods 6010A and 200.7 provided 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.
- 1.3. Method 6010A 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 do require digestion of dissolved samples and this must be clarified before project initiation.
- 1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable and total elements in surface water, domestic and industrial waste waters. 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.
- 1.5. This SOP is not applicable to the analysis of drinking water samples due to the wide array of state specific requirements which must be accommodated. Refer to facility specific SOPs for guidance on performing drinking water analyses.

2. SUMMARY OF METHOD

- 2.1. This method describes a technique for the determination of elements in solution. 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 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 where a background corrective measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken.

- 2.2. Consult the appropriate SOP's 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. Interelement correction factors (IEC's) are necessary to compensate for spectral overlap. Interelement 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. Interelement corrections (IEC's) must be applied to the analyte to remove the effects of these unwanted emissions. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."
- 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, 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 acid is used in cleaning the ICP torch and hydrofluoric acid is commonly used in air toxics preparations.)
- 5.3.2. The following materials are known to be **oxidizing agents**:
nitric acid and hydrogen peroxide.
- 5.3.3. The plasma emits strong UV light and is harmful to vision. 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 with the sash closed as far as the operation will permit.
- 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. Nitrogen or 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 multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously 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.
- 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.
- 8.3. Soil samples do not require preservation but must be stored at $4^{\circ}\text{C} \pm 2^{\circ}$ until the time of analysis.

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010A, the following requirements must be met.

- 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 any way 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/matrix prior to the analysis of any 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. MDL's 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 valid. The result of the MDL determination must be below the Quanterra reporting limit.
- 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 standards used to define the linear range limit must be analyzed during a

routine analytical run. The determined concentration of the linear range standard must be within 5% of the true value. The linear range is the concentration above which results cannot be reported without dilution of the sample. If the instrument is adjusted in any way that may affect the LR's, the LR's must be redetermined.

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. Background correction points must be set prior to determining IEC's. 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. Interelement Corrections (IEC) - ICP interelement correction factors must be determined prior to the analysis of samples and annually thereafter. If the instrument is adjusted in any way that may affect the IEC's, the IEC's must be redetermined. When initially determining IEC's 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 IEC's. 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 \pm the RL as defined in Tables I, IA or II.

Note: Trace ICP IEC's are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IEC's will be required on a more frequent basis for the Trace as reflected by the ICSA response.

9.1.6. Rinse Time Determination - 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). 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, a control limit of 80 - 120% 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% 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 case 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/MSD's. 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. Control limits of 80 - 120 % recovery and 20% RPD 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. Serial Dilution (L) - Serial dilution analysis 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 serial dilution sample. The serial dilution is performed by running a sample at a 4x dilution. Samples identified as field blanks cannot be used for serial dilution analyses. 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 40x the IDL. If the results are not within 10%, narrate the possibility of chemical or physical interference.
- 9.6. High Calibration Standard (HCAL) - At the beginning of the run, prior to the analysis of samples, the high standard must be rerun and recovered within 95 - 105%. If any analyte of interest falls outside the acceptance criteria corrective action must occur. The analysis should be terminated, the problem resolved and the instrument recalibrated. (See Section 11.11 or 11.12 for required run sequence.)
- 9.7. 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. For Method 6010A, the ICV must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (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).
- 9.8. 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 must be a mid-range standard made from a 2x dilution of the

calibration standard. The CCV result must fall within 10% of the true value for that solution. 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. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis 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).

- 9.9. 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 interferences. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom Quanterra multielement ICS solutions must be used. All analytes must be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it must be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. Elements known to be interferences 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.
- 9.9.1. The ICSA and ICSAB solutions must be run at the beginning and end of the run or every 8 hours, whichever is more frequent. (See Section 11.11 or 11.12 for required run sequence.)
- 9.9.2. The ICSAB results for the interferences must fall within 80 - 120% of the true value. If any ICSAB interference result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.
- 9.9.3. The ICSAB analytes must be recovered within 80 - 120% of the true value. If the ICSAB analytes do not meet criteria the analysis must be terminated, the problem corrected, the instrument recalibrated and the samples rerun for the affected analytes.
- 9.9.4. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the Quanterra guidelines of +/- 2x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL 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 $\pm 2x$ 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 $\pm 2x$ 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 IEC's 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.
- 9.10. CRI - To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run following the ICB. The CRI standard must contain all analytes at *no greater than two times the standard RL*. The CRI results must fall within 50% of the true value *and the problem must be corrected before continuing with the analysis. When the sample RL is greater than the CRI true value, a standard other than the CRI may be used to evaluate linearity near the RL.*
- 9.11. 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.
- 9.12. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these 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. Calibrate the instrument according to the instrument manufacturer's recommendations. Refer to the facility specific instrument SOP and ICP instrument manual for 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 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 samples must be digested unless it can be documented that the sample meets all of the following criteria:
 - A. COD is < 20 ppm.
 - B. Visibly transparent with a turbidity measurement of 1 NTU or less.
 - C. Colorless with no perceptible order.
 - D. Is of one liquid phase and free of particulate or suspended matter following acidification.
- 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 and post digestion spikes 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 non-Trace ICP's.
- 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	5
SW846 3005	2	5
SW846 3010	3	5

The following procedural guidelines must be followed when using an internal standard:

- 11.10.1. Recommended 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 screened without the addition of the internal standard.
- 11.10.5.3. If the internal standard element is not identified in the unspiked field sample at a level exceeding 10% of the level spiked, the data may be accepted.
- 11.10.5.4. If the IS analyte is detected in the unspiked field sample at a concentration greater than 10% of the spiked level then either:
- A different internal standard must be used.
 - The IS concentration must be raised.
 - The sample must be diluted and rerun.
 - The analysis must be run without an internal standard (matrix matching must be substituted.)

11.11. The following analytical sequence must be used for Methods 6010A and 200.7:

Instrument Calibration

ICV

ICB

CRI

HCAL

ICSA

ICSAB

6 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
ICSA
ICSAB
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010A and 200.7 quality control criteria.

- 11.12. The following run sequence is consistent with 200.7, 6010A and CLP requirements and may be used as an alternate to the sequence specified in 11.11 if multiple methods must be accommodated in the same analytical run:

Instrument Calibration

ICV
ICB
CRI
HCAL
ICSA
ICSAB
CCV
CCB
10 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
CRI
ICSA
ICSAB
CCV
CCB

Refer to the CLP SOP (CORP-MT-0002) for additional quality control requirements.

11.13. The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 9.8:

Original Run: Instrument Calibration

ICV
ICB
CRI
HCAL
ICSA
ICSAB
6 samples
CCV1
CCB1
10 samples
CCV2
CCB2
10 samples **
CCV3 * * Failure occurs at CCV3/CCB3
CCB3 * **Samples requiring rerun for affected analytes
10 samples **
CCV4
CCB4
10 samples
CCV5
CCB5
ICSA
ICSAB
CCV6
CCB6

Reanalysis: Recalibrate

ICV
ICB
CRI
HCAL
CCV2
CCB2
10 samples
CCV3
CCB3
10 samples
CCV4
CCB4
ICSA
ICSAB

CCV6
CCB6

Notes: If the reanalysis is conducted under the same instrument setup conditions then it is not necessary to rerun the ICSA/ICSAB at the start of the reanalysis sequence as long as the 8 hour criteria are met. If reanalysis can't be initiated immediately or under the same run conditions then reanalysis must be conducted using the full analysis sequence as detailed in Section 11.11.

Samples between CCV4 and CCV5 do not require reanalysis as they were bracketed by compliant QC samples.

See CORP-MT-0002 for the appropriate reanalysis sequence if CLP requirements must also be met.

- 11.14. Full method required QC must be available for each wavelength used in determining reported analyte results.
- 11.15. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.
- 11.16. 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 is 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.
- 11.17. 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 D provides guidance on performing MSA analyses.

- 11.18. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix.

radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

- 11.19. 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:

$$\%R = 100 \left(\frac{\text{Found(ICV)}}{\text{True(ICV)}} \right)$$

- 12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \left(\frac{\text{Found(CCV)}}{\text{True(CCV)}} \right)$$

- 12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \left(\frac{\text{SSR} - \text{SR}}{\text{SA}} \right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

- 12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100 \left[\frac{|\text{MSD} - \text{MS}|}{\left(\frac{\text{MSD} + \text{MS}}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

$$RPD = 100 \left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2} \right)} \right]$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg / L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight in Kg of wet sample digested

S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \left(\frac{\text{Found (LCS)}}{\text{True (LCS)}} \right)$$

- 12.8. The serial dilution percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)

S = Serial dilution result (Instrument reading × 4)

- 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 6010A 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 matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value until in house control limits are established.
- 13.4. Training Qualification:

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 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 according to the facility hazardous waste procedures. 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. *Methods for the Determination of Metals in Environmental Samples, EPA/600/R-94/111. Supplement I, Revision 4.4, May 1994. Method 200.7.*
- 16.2. 40 CFR Part 136, Table IB, 7-1-92.
- 16.3. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846. 3rd Edition. Final Update I, July 1992. Method 6010A.
- 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 from both Method 6010A and 200.7.

- 17.1.1.1. Method 200.7 and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

- 17.1.1.2. The alternate run sequence presented in Section 11.12 is consistent with method requirements. Additional QC analyses were added to accommodate the CLP protocol requirements.

17.1.1.3. The calibration blank is prepared in an acid matrix of 5% HNO₃/5% HCl instead of the specified 2% HNO₃/10% 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. Modifications from Method 200.7.

17.1.2.1. Blank subtraction is not performed as per Quanterra QC policy. Method blank results are provided in the analytical report.

17.1.2.2. 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 a reagent blank signal. Quanterra labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.3. Section 7.6.3 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.

17.1.2.4. The ICS criteria applied by this SOP differ from those stated in the method. 200.7 states that results should fall within the established control limits of 1.5 times the standard deviation of the mean value. These control criteria were based on a specific solution made available by EMSL-Cincinnati to labs several years ago. Since this solution is no longer available, Quanterra has modeled their ICSA/ICSAB solutions on the design of the ICSA/ICSAB solution provided by EPA directly to EPA contract laboratories. The control limits listed in this SOP are those which EPA states applicable to the EPA designed solution.

17.1.2.5. Method 200.7 states the CCB should be within 2x the standard deviation of the average blank reading. The intent of this requirement is to ensure that the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria.

17.1.3. Modifications from Method 6010A.

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. Calibration is performed according to instrument manufacturer's recommendations. Method 6010A provides contradictory instructions for instrument calibration by stating to calibrate both according to instrument manufacturer's recommendations as well as to use a blank and three standards. TJA has stated that the use of multiple standards may be detrimental to determinations near the detection limit due to the inability of the linear equation to force-fit through the origin (see Appendix C) and recommends the calibration be performed using a blank and one standard. Leeman recommends a blank and three standards. EPA has stated that manufacturer's recommendations should take precedence and that the next version of the method, 6010B, will clear up the issue by stating "The calibration curve should consist of a minimum of a blank and a standard." This SOP requires verification of the initial instrument calibration using a CRI at two times the RL, an ICV at 5 - 25% of the calibration level, a CCV at 50% of the calibration level and by rerunning the high calibration standard (HCAL) post calibration to demonstrate linearity (See Tables I, IA and II).

17.1.3.3. Section 5.6 of 6010A states that the instrument check standard (CCV) should be prepared from a second source standard. This SOP states that the CCV will be from the same source as the calibration standards. The purpose of the second source standard is to verify the accuracy of the calibration standards. The intent of this requirement is met through the analysis of a second source ICV standard prior to the analysis of samples. The use of a same source CCV provides for a more accurate and consistent measure of instrument drift from initial calibration.

17.1.3.4. Section 5.7 states that spiking of the ICS solution with analytes is not required if the ICP will display overcorrection as a negative number. All Quanterra instrumentation has this capability and therefore the spike analysis is not required. Quanterra does run a spiked ICSAB but the analytes are not spiked at the 10x IDL level referenced in 6010A. The ICSAB solution run by Quanterra is based

on the design of the ICSA/ICSAB solution provided by EPA directly to contract environmental labs.

17.1.3.5. Method 6010A uses a Quality Control Standard (QCS) on a weekly basis to verify calibration standard accuracy. Quanterra refers to the QCS as an ICV and the accuracy verification is performed on a daily basis. The QCS described in Method 6010A is made to contain analytes at 10x the IDL. The Quanterra ICV solution is not made at 10x IDL for all elements as this concentration is not appropriate relative to the standard reporting limits. Quanterra designed the ICV to be a reliable indicator of calibration standard accuracy by raising the analyte concentrations to a level where the analytical determination is not impacted by low level curve bias.

17.1.3.6. Method 6010A states the CCB should be within 3x the standard deviations of the average blank reading. The intent of this requirement is to ensure that the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria.

17.2. Modifications from previous SOP

None.

17.3. Facility Specific SOP's

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. Refer to the appendices for any facility specific information required to support this SOP.

17.3.1. Refer to the SOP change form on file in North Canton's Quality Assurance department.

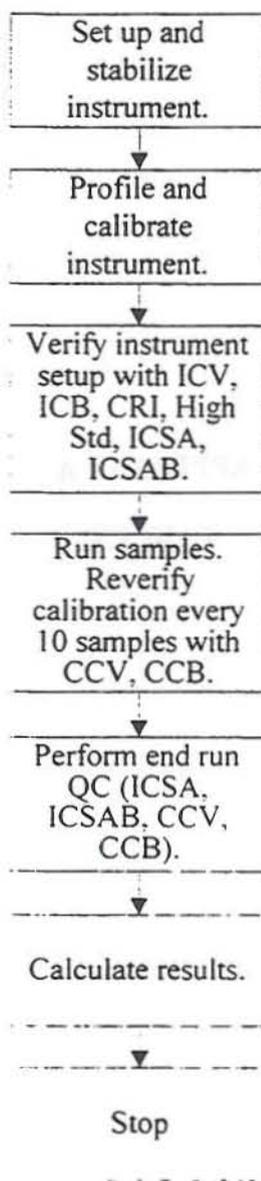
17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout signed by analyst).
- 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 date, source and lot #).
- Non-conformance summary (if applicable).

17.5. Flow Diagram



APPENDIX A
TABLES

TABLE I. Method 200.7 and 6010A Analyte List

ELEMENT	Symbol	CAS #	6010A 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	7439-93-1	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		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	50
Silver	Ag	7440-22-2	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-28-0	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 6010A Trace ICP 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	7440-03-26	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)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	400	25000	50000
Antimony	10000	60	120	1000	5000
Arsenic	10000	300	600	1000	5000
Barium	10000	200	20	1000	5000
Beryllium	10000	5	10	1000	5000
Cadmium	10000	5	10	1000	5000
Calcium	100000	5000	10000	25000	50000
Chromium	10000	10	20	1000	5000
Cobalt	10000	50	100	1000	5000
Copper	10000	25	20	1000	5000
Iron	100000	100	100	25000	50000
Lead	10000	100	200	1000	5000
Lithium	10000	50	100	1000	5000
Magnesium	100000	5000	10000	25000	50000
Manganese	10000	15	20	1000	5000
Molybdenum	10000	40	80	1000	5000
Nickel	10000	40	80	1000	5000
Phosphorous	10000	300	600	1000	5000
Potassium	100000	5000	10000	25000	50000
Selenium	10000	250	500	1000	5000
Silver	2000	10	20	500	1000
Sodium	100000	5000	10000	25000	50000
Strontium	10000	50	100	1000	5000
Thallium	20000	2000	4000	5000	10000
Vanadium	10000	50	100	1000	5000
Zinc	10000	20	40	1000	5000
Boron	10000	200	400	1000	5000
Silicon	10000	500	1000	1000	5000
Tin	10000	100	200	1000	5000
Titanium	10000	50	100	1000	5000
Bismuth	10000	200	400	1000	5000
Zirconium	10000	100	200	1000	5000
Tellurium	10000	500	1000	1000	5000
Thorium	10000	500	1000	1000	5000
Uranium	10000	500	1000	1000	5000
Tungsten	10000	500	1000	1000	5000
Palladium	10000	100	200	1000	5000

TABLE IVA. Trace Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	100	12500	25000
Antimony	1000	10	10	250	500
Arsenic	1000	10	10	250	500
Barium	4000	10	20	1000	2000
Beryllium	4000	5	10	1000	2000
Cadmium	1000	2	2	250	500
Calcium	100000	5000	10000	25000	50000
Chromium	4000	5	10	1000	2000
Cobalt	4000	50	40	1000	2000
Copper	4000	25	20	1000	2000
Iron	50000	100	100	12500	25000
Lead	1000	3	6	250	500
Magnesium	100000	5000	10000	25000	50000
Manganese	4000	15	20	1000	2000
Molybdenum	4000	40	20	1000	2000
Nickel	4000	40	80	1000	2000
Potassium	100000	5000	10000	25000	50000
Selenium	1000	5	10	250	500
Silver	2000	5	10	500	1000
Sodium	100000	5000	10000	25000	50000
Thallium	2000	10	20	500	1000
Vanadium	4000	50	40	1000	2000
Zinc	4000	20	40	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 must be spiked into the ICSAB at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (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
Reanalysis of High Standard (HCAL)	Beginning of every analytical run, after CRI.	95 - 105 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010A: 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.	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 and end of every run and every 8 hours.	Analyte results must be within +/- 2x RL from zero for analytes with RL ≤ 10 ug/L.	See Section 9.9.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.9.
CRI	Immediately following ICB.	Results must be within 50 - 150% recovery.	Terminate analysis; Correct the problem; Recalibrate.

* 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
Serial Dilution	One per prep batch.	For samples > 40x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference.
Method Blank	One per sample preparation batch of up to 20 samples.	<p>The result must be less than or equal to the RL.</p> <p>Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2).</p> <p>Sample results greater than 20x the blank concentration are acceptable.</p> <p>Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).</p>	<p>Redigest and reanalyze samples.</p> <p>Note exceptions under criteria section.</p> <p>See Section 9.2 for additional requirements.</p>
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	<p>Aqueous LCS must be within 80 - 120% recovery or in-house control limits.</p> <p>Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.3)</p>	<p>Terminate analysis;</p> <p>Correct the problem;</p> <p>Redigest and reanalyze all samples associated with the LCS.</p>
Matrix Spike	One per sample preparation batch of up to 20 samples.	80 - 120 % recovery. If the MS/MSD is out for an analyte, it must be in control in the LCS. For TCLP See Section 11.17.	<p>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.</p>

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE
ELEMENT ANALYSIS, METHOD 6010A AND METHOD 200.7
APPENDIX A - TABLES

SOP No. CORP-MT-0001NC
Revision No. 1.2
Revision Date: 04/17/97
Page: 40 of 52

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike Duplicate	See Matrix Spike	80 - 120 % recovery; RPD \leq 20% (See MS).	See Corrective Action for Matrix Spike.

Item	Yes	No	Comments
1. All data points are within the acceptable range.			
2. The standard deviation is within the acceptable range.			
3. The correlation coefficient is within the acceptable range.			
4. The detection limit is within the acceptable range.			
5. The quantification limit is within the acceptable range.			
6. The recovery is within the acceptable range.			
7. The precision is within the acceptable range.			
8. The accuracy is within the acceptable range.			
9. The stability is within the acceptable range.			
10. The repeatability is within the acceptable range.			
11. The reproducibility is within the acceptable range.			
12. The linearity is within the acceptable range.			
13. The sensitivity is within the acceptable range.			
14. The selectivity is within the acceptable range.			
15. The robustness is within the acceptable range.			
16. The ruggedness is within the acceptable range.			
17. The reliability is within the acceptable range.			
18. The validity is within the acceptable range.			
19. The consistency is within the acceptable range.			
20. The comparability is within the acceptable range.			

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: 6010A / 200.7: CORP-MT-0001 Rev 1
 CLP : 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 ? (ICV: 6010A, CLP = 90 - 110%, 200.7 = 95 -105%) (CCV: 90 - 110%)				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. High Std. (HCAL) reanalyzed before samples and recovered within QC limits ? (6010A/200.7 95-105%, CLP- N/A)				
5. CRI run and recovered within QC limits ? (+/- 50% for non-CLP)				
6. 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. Serial dilution 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: _____

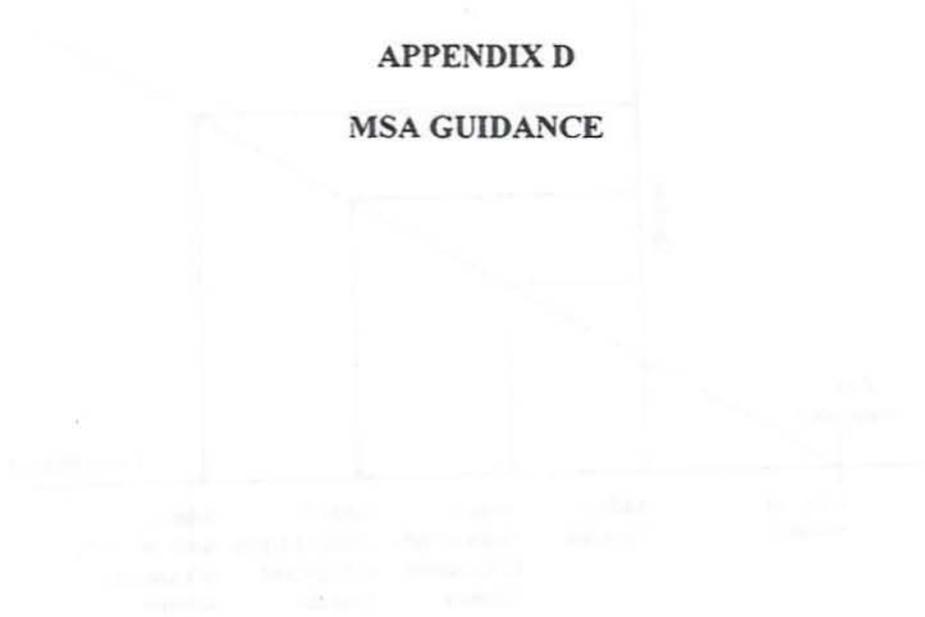
APPENDIX C

TJA CALIBRATION RECOMMENDATION

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE
ELEMENT ANALYSIS, METHOD 6010A AND METHOD 200.7
APPENDIX C- TJA CALIBRATION RECOMMENDATION

SOP No. CORP-MT-0001NC
Revision No. 1.2
Revision Date: 04/17/97
Page: 44 of 52

APPENDIX D
MSA GUIDANCE

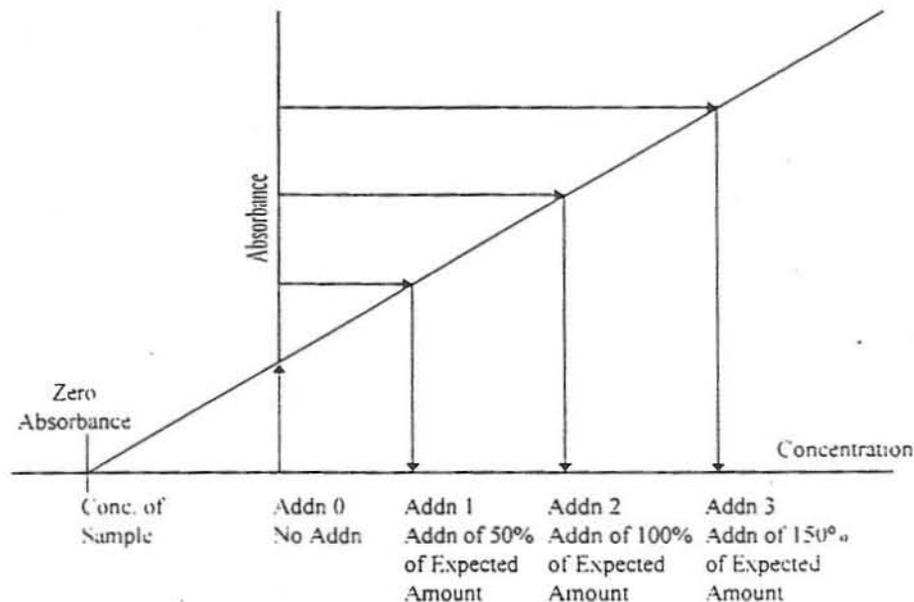


Appendix D. MSA Guidance

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

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 IEC's
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

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)

Controlled Copy
Copy No. _____

UNCONTROLLED COPY

SOP No. CORP-MT-0005NC
Revision No. 1.1
Revision Date: 04-19-97
Page: 1 of 40

QUANTERRA STANDARD OPERATING PROCEDURE

**TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY
COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1**

(SUPERSEDES: REVISION 0)

Prepared by: *Phil Rine* 4/16/97
Principal Scientist, Inorganics

Reviewed by: *Phil Rine* 4/22/97
Technology Standardization Committee

Approved by: *Paul H. [Signature]* 4/23/97
Director, Quality Assurance

Approved by: *Henry J. [Signature]* 4-23-97
Director, Environmental Health and Safety

Approved by: *Christy R. [Signature]* 4-23-97
Management

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

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	8
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	9
9. QUALITY CONTROL.....	10
10. CALIBRATION AND STANDARDIZATION	15
11. PROCEDURE	14
12. DATA ANALYSIS AND CALCULATIONS.....	18
13. METHOD PERFORMANCE.....	20
14. POLLUTION PREVENTION	20
15. WASTE MANAGEMENT	20
16. REFERENCES	21
17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .).....	21

LIST OF APPENDICES:

APPENDIX A - TABLES	26
APPENDIX B - QUANTERRA Hg DATA REVIEW CHECKLIST	30
APPENDIX C - MSA GUIDANCE	32
APPENDIX D - TROUBLESHOOTING GUIDE	35
APPENDIX E - CONTAMINATION CONTROL GUIDELINES	37
APPENDIX F - PREVENTATIVE MAINTENANCE	39

1. SCOPE AND APPLICATION

- 1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1.
- 1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.
- 1.3. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed, however Method 7471A (see CORP-MT-0007) is usually the method of choice. All matrices require sample preparation prior to analysis.
- 1.4. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, domestic and industrial wastes. All matrices require sample preparation prior to analysis.
- 1.5. The Quanterra reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP, SPLP or EPTOX leachates for which the reporting limit is 0.002 mg/L.

2. SUMMARY OF METHOD

- 2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. 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 digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

- 4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.
- 4.2. Copper has also been reported to interfere: however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.
- 4.3. Chlorides can cause a positive interference. Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample head space before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

- 4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the

sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

- 4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.
- 4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

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. The following specific hazards are known:
 - 5.3.1. The following materials are known to be **corrosive**:
hydrochloric acid, nitric acid and sulfuric acid.
 - 5.3.2. The following materials are known to be **oxidizing agents**:
nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.
 - 5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked

for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M KMnO_4 and 10% H_2SO_4 , or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

- 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. 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 with the sash closed as far as the operation will permit.
- 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. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.
- 5.8. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.
- 5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

- 6.1. Temperature controlled water bath (capable of maintaining a temperature of 90-95 °C) or autoclave that is able to obtain conditions of 15 lbs., 120 °C for 15 minutes.

- 6.2. Atomic Absorption Spectrophotometer equipped with:
- 6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.
 - 6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).
 - 6.2.3. Peristaltic pump which can deliver 1 L/min air.
 - 6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.
 - 6.2.5. Recorder or Printer.
 - 6.2.6. Aeration Tubing: A straight glass frit having a coarse porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.
 - 6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that achieve the same purpose are also acceptable (i.e., Gortex filter).
- NOTE:** Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.
- 6.3. BOD bottles or equivalent.
 - 6.4. Nitrogen or argon gas supply, welding grade or equivalent.
 - 6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.
 - 6.6. Class A volumetric flasks.
 - 6.7. Thermometer (capable of accurate readings at 95 °C).
 - 6.8. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

- 7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
- 7.2. Stock (1000 ppm) mercury standards (in 10% HNO₃) are purchased as custom Quanterra solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.
- 7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO₃. This acid (2 mL of concentrated HNO₃) must be added to the flask/bottle before the addition of the stock standard aliquot.
- 7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO₃. This acid (150 uL of concentrated HNO₃) must be added to the flask/bottle before the addition of the stock standard aliquot.
- 7.5. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working mercury standard into 100 mL flasks and diluting to volume with reagent water.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.
- 7.6. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.
- 7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All

standards must be processed through the entire analytical procedure including sample preparation.

- 7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

- 7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

- 7.10. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

- 7.11. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

- 7.12. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

- 7.13. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Sample holding time for mercury is 28 days 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. Refrigeration is not required. Preservation must be verified prior to analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7470A or the 245.1, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any 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. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a 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 pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS) are not included in the sample count for determining the size of a preparation batch. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.

9.4. 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 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 20 times higher than the blank contamination level).

- 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.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. 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. The LCS must be carried through the entire analytical procedure. The CCV results can be reported as LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.
- In the instance where the LCS recovery is $> 120\%$ 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 case narrative.

- 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. ...
 - Corrective action will be re-preparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.
- 9.6. 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/MSD's. 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 Table I (Appendix A).
- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD 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 re-preparation 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 4 times 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.
- 9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of

the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.11 and Section 11.2.12 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

- 9.8. 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 must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.11 and 11.2.12 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.
- 9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. 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 11.2.13 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.
- 10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.
- 10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

- 10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.
- 10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the Quanterra reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.
- 10.6. The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.
- 10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Sample Preparation:

- 11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.
- 11.1.2. Transfer 100 mL of well mixed sample or standard to a clean sample digestion bottle.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

- 11.1.3. Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 , mixing after each addition.

Note: All spiking should be done after the initial addition of acids.

- 11.1.4. Add 15 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple

color persists for at least 15 minutes. If after the addition of up to 25 mL additional permanganate the color does not persist, sample dilution prior to reanalysis may be required.

Note: When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all other associated samples and standards in the run. In instances, where this is not feasible, the addition of excess reagent can be addressed through mathematical correction of the results to account for the resultant dilution effect.

11.1.5. Add 8 mL of potassium persulfate solution and heat for two hours in a water bath at 90 - 95 °C.

NOTE: Alternatively, for RCRA analyses using 7470A, samples may be digested using an autoclave for 15 minutes at 120 °C and 15 lbs.

11.1.6. Cool samples.

11.2. Sample Analysis:

- 11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.
- 11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.
- 11.2.3. When ready to begin analysis, add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Add this solution in 6 mL increments until the permanganate is completely reduced.

- 11.2.4. Manual determination:
 - 11.2.4.1. Treating each sample individually, purge the head space of the sample bottle for at least one minute.
 - 11.2.4.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.
 - 11.2.4.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.
 - 11.2.4.4. Place the aeration device into 100 mL of 1% HNO₃ and allow to bubble rinse until the next sample is analyzed.
- 11.2.5. Automated determination: Follow instructions provided by instrument manufacturer.
- 11.2.6. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.
- 11.2.7. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.
- 11.2.8. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.
- 11.2.9. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.10. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.11. The following analytical sequence must be used with 7470A and 245.1:

Instrument Calibration

ICV

ICB

Maximum 10 samples

CCV

CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run

CCV

CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7470A and 245.1.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.12. The following run sequence is consistent with 7470A, CLP and 245.1 and may be used as an alternate to the sequence in 11.2.11. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration

ICV

ICB

CRA*

CCV

CCB

10 samples

CCV

CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.

CCV

CCB

Refer to the appropriate CLP SOP (CORP-MT-0006) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.13. 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 matrix spike levels for TCLP analyses are detailed in Table I (Appendix A). Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

- 11.3. 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.
- 11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards. preventive maintenance and troubleshooting.
- 11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix. radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- 11.6. 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:

$$\%R = 100 \left(\frac{\text{Found}(ICV)}{\text{True}(ICV)} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \left(\frac{\text{Found}(CCV)}{\text{True}(CCV)} \right)$$

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

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100 \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

$$RPD = 100 \left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2} \right)} \right]$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

- 12.6. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \left(\frac{\text{Found}(LCS)}{\text{True}(LCS)} \right)$$

- 12.7. Appropriate factors must be applied to sample values if dilutions are performed.
- 12.8. 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. Method performance is determined by the analysis of method blanks, laboratory control samples, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The laboratory control sample should recover within 20% of the true value until in house limits are established.
- 13.3. Training Qualification:

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 experience.

14. POLLUTION PREVENTION

- 14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

- 15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

- 16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).
- 16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.1.
- 16.3. U.S.EPA Statement of Work for Inorganics Analysis; ILMO3.0.
- 16.4. QA-003, Quanterra QC Program.
- 16.5. QA-004, Rounding and Significant Figures.
- 16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

- 17.1. Modifications/Interpretations from reference method.
 - 17.1.1. Modifications from both 7470A and 245.1.
 - 17.1.1.1. The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
 - 17.1.1.2. This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V. "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."
 - 17.1.1.3. The alternate run sequence presented in Section 11.2.12 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7470A

- 17.1.2.1. 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.
- 17.1.2.2. Documentation is on file from EPA's Office of Solid Waste (Olliver Fordham 11/28/95) regarding the acceptance of the autoclave as an equivalent heating device to the water bath. In his letter, Mr. Fordham cited the CLP water protocol 245.1 CLP-M and therefore the operating parameters from that method were adopted for 7470A (15 minutes at 120 °C and 15 lbs.).
- 17.1.2.3. Method 7470A does not state control criteria within the text of the method. The QC section of 7470A refers the analyst to Section 8.0 of Method 7000A, the generic atomic absorption method, which discusses flame and furnace methods. The ICV criteria stated in Method 7000A is $\pm 10\%$. This SOP requires ICV control limits of $\pm 20\%$ based on the fact that the mercury ICV, unlike the ICV for the flame and furnace analytes, is digested and therefore is equivalent to a LCS. The CLP protocol 245.1 CLP-M recognizes this factor and requires control limits of $\pm 20\%$.

17.1.3. Modifications from 245.1

- 17.1.3.1. Method 245.1 Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. Quanterra reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

None.

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. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Aqueous Sample Preparation - Mercury

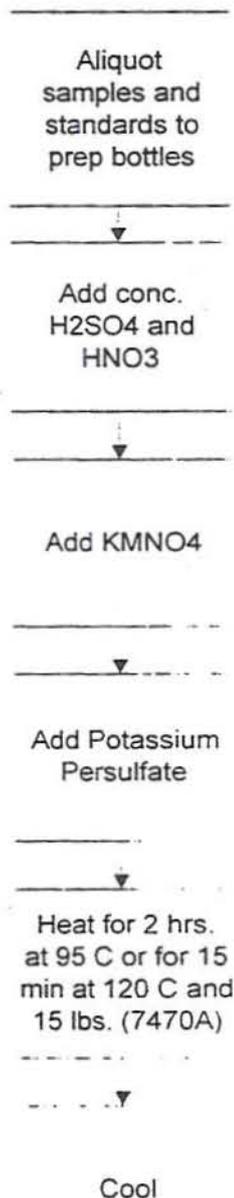
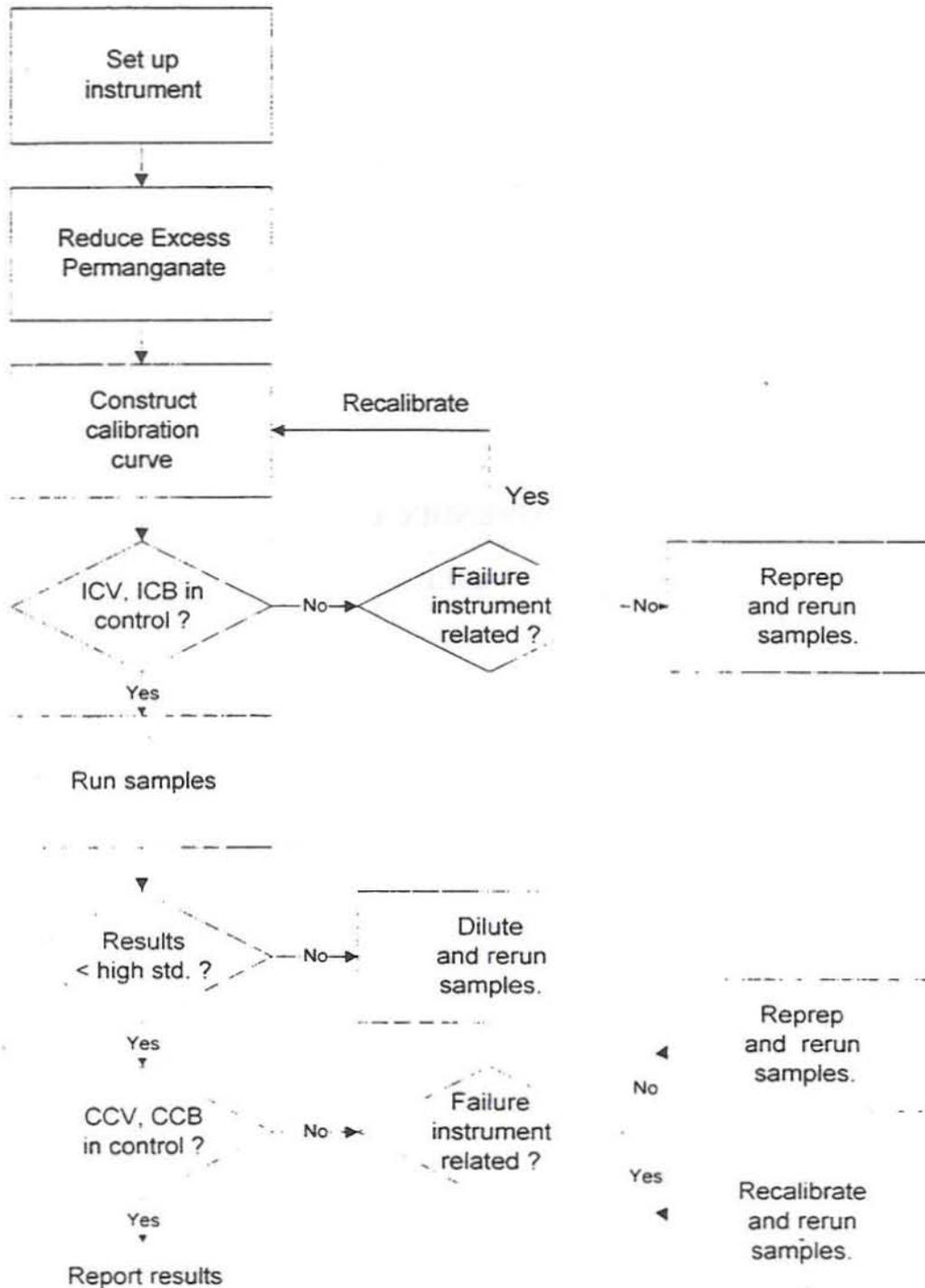


Figure 2. CVAA Mercury Analysis



APPENDIX A
TABLES

**TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC
 STANDARD AND SPIKING LEVELS (MG/L)**

Standard Aqueous RL	0.0002
TCLP RL	0.002
Std 0	0
Std 1	0.0002
Std 2	0.0005
Std 3	0.001
Std 4	0.002
Std 5	0.005
Std 6 **	0.010
ICV	0.001 or 0.0025 ***
LCS/CCV	0.0025 or 0.005 ***
Aqueous MS	0.001
TCLP MS	0.005

- * SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.
- ** Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.
- *** Concentration level dependent on high calibration standard used. CCV must be 50% of high standard concentration and ICV must be 20-25% of high standard concentration.

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90-110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.8).
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 or reprep batch (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.11 and 11.2.12 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
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.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data: no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.2.13
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits: RPD \leq 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
QUANTERRA Hg DATA REVIEW CHECKLIST

Quanterra Hg Data Review Checklist

Run/Project Information

Run Date: _____ Analyst: _____ Instrument: _____
 Prep Batches Run: _____

Circle Methods used: 7470A / 245.1 : CORP-MT-0005 Rev 1 7471 / 245.5 : CORP-MT-0007 Rev 1
 CLP - AQ : CORP-MT-0006 Rev 0 CLP - SOL : CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard 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?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ Date: _____
 Comments: _____

2nd Level Reviewer : _____ Date: _____

APPENDIX C
MSA GUIDANCE

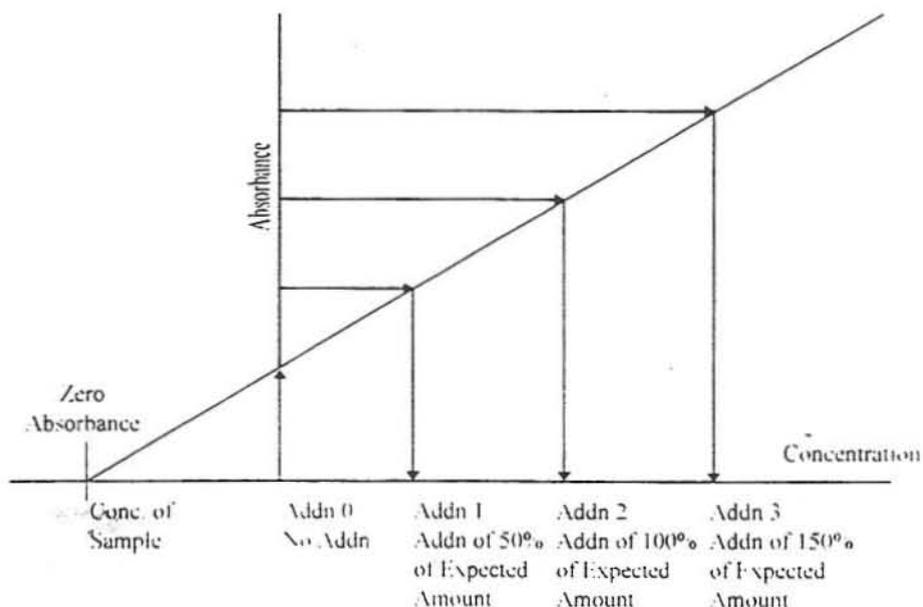
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where $y=0$) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Problem	Cause	Solution
Low sensitivity	Mercury lamp not on	Turn on mercury lamp
High background	Contaminated reagents	Use high purity reagents
Low signal	Sample volume too small	Increase sample volume
High signal	Sample volume too large	Decrease sample volume
No signal	Mercury lamp out of alignment	Align mercury lamp
Fluctuating signal	Unstable power supply	Check power supply
High noise	Dirty cuvette	Clean cuvette
Low signal	Expired reagents	Use fresh reagents
High signal	Contaminated cuvette	Use clean cuvette
No signal	Mercury lamp not on	Turn on mercury lamp
Low signal	Sample volume too small	Increase sample volume
High signal	Sample volume too large	Decrease sample volume
No signal	Mercury lamp out of alignment	Align mercury lamp
Fluctuating signal	Unstable power supply	Check power supply
High noise	Dirty cuvette	Clean cuvette
Low signal	Expired reagents	Use fresh reagents
High signal	Contaminated cuvette	Use clean cuvette

APPENDIX D
TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E
CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F. 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.

Cold Vapor Atomic Absorption (Leeman PS 200)⁽¹⁾

Daily	Semi-annually	Annually
Clean lens.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000)⁽¹⁾

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

Controlled Copy

Copy No. _____

Implementation Date _____

UNCONTROLLED COPY

SOP No. NC-WC-0084

Revision No: 2

Revision Date: 02/11/99

Page 1 of 22

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY

(SUPERSEDES: REVISION (1))

Prepared by:	<u>Mark R. Bane</u>	<u>2/12/99</u>	Date
Reviewed by:	<u>Jessica K. Roberts</u>	<u>2/12/99</u>	Date
	Technology Specialist		
Approved by:	<u>Quilley Lee</u>	<u>2-11-99</u>	Date
	Quality Assurance Manager		
Approved by:	<u>[Signature]</u>	<u>2-17-99</u>	Date
	Environmental Health and Safety Coordinator		
Approved by:	<u>Christy McPhee</u>	<u>2-18-99</u>	Date
	Laboratory Director		

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held propriety to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any propose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be give to those parties unless those parties also specifically agree to these conditions

1. SCOPE AND APPLICATION.....	3
2. SUMMARY OF METHOD	3
3. DEFINITIONS	3
4. INTERFERENCES	3
5. SAFETY.....	4
6. EQUIPMENT AND SUPPLIES	5
7. REAGENTS AND STANDARDS.....	5
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	11
9. QUALITY CONTROL.....	12
10. CALIBRATION AND STANDARDIZATION	14
11. PROCEDURE	15
12. DATA ANALYSIS AND CALCULATIONS	16
13. METHOD PERFORMANCE	16
14. POLLUTION PREVENTION	17
15. WASTE MANAGEMENT	17
16. REFERENCES.....	17
17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)	18

1. SCOPE AND APPLICATION

- 1.1. This method covers the determination of fluoride, chloride, nitrite, bromide, nitrate, ortho-phosphate and sulfate in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction 11.7) and leachates (when no acetic acid is used).

2. SUMMARY OF METHOD

- 2.1. A 25 uL volume of sample is introduced into the ion chromatograph. The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn or guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppresser column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3. DEFINITIONS

- 3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

- 4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.
- 4.2. The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of concentrated eluent to each standard and sample.
- 4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

-
- 4.4. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.
- 4.5. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

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 satisfied 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 chemicals are known to be highly corrosive:
- sulfuric acid
- 5.4. 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 when possible. 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 with the sash closed as far as the operation will permit.
- 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.
- 6.2. Ion Chromatograph -- Analytical system complete with ion chromatograph and all required accessories including analytical columns, compressed gases and detectors.
 - 6.2.1. Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with same substrate as the separator column. 4 x 50 mm, Dionex AG4A-SC P/N 43175, or equivalent.
 - 6.2.2. Anion separator column: The separation shown in Figure 1 was generated using a Dionex AS4A-SC column (P/N 43174). Equivalent column may be used if comparable resolution is obtained, and the requirements of Sect. 9.2 can be met.
 - 6.2.3. Anion suppresser device: Dionex anion micro membrane suppresser (P/N 37106) or equivalent.
 - 6.2.4. Detector -- Conductivity cell: approximately 1.25 uL internal volume, Dionex, or equivalent.
 - 6.2.5. Dionex AI - 450 Data Chromatography Software or equivalent.
- 6.3. Assorted laboratory glassware (pipettes, volumetric flasks, etc.).

7. REAGENTS AND STANDARDS

- 7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 7.2. Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

-
- 7.3. Eluent solution: sodium bicarbonate (CASRN 144-55-8) 1.7 mM, sodium carbonate (CASRN 497-19-8) 1.8 mM. Dissolve 2.856 g sodium bicarbonate (NaHCO_3) and 3.816 g of sodium carbonate (Na_2CO_3) in reagent water (7.2) and dilute to 100 mL in a volumetric flask. Take 10 mL of this concentrated eluent solution and dilute to 2 L for use as the working eluent solution or dissolve the entire bicarbonate/carbonate amount in 20 L of reagent water.
- 7.4. Regeneration solution (micro membrane suppresser): Sulfuric acid (CASRN-7664-93-9) 0.025N. Dilute 2.8 mL concentrated sulfuric acid (H_2SO_4) to 4 L with reagent water.
- 7.5. Stock solutions (1,000 mg/L): All stocks may be prepared as described below or purchased from commercial sources. Primary and secondary sources are required for each target analyte.
- 7.5.1. Fluoride stock solution (1.00 mL = 1.00 mg F^-): In a 1 liter volumetric flask, dissolve 2.2100 g of sodium fluoride (NaF) in reagent water, and dilute to volume with reagent water. Store in chemical- resistant glass or polyethylene.
- 7.5.2. Chloride stock solution (1.00 mL = 1.00 mg Cl^-): Dry sodium chloride (NaCl) for 12 hours at 105°C , and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.6485 g of the dry salt in reagent water and dilute to volume with reagent water.
- 7.5.3. Nitrite stock solution (1.00 mL = 1.00 mg $\text{NO}_2^- - \text{N}$): Place approximately 10.0 g of sodium nitrite (KNO_2) in a 125 mL beaker and dry to constant weight (about 24 hours) in a desiccator. In a 1 liter volumetric flask, dissolve 6.0790 g of the dried salt in reagent water and dilute to volume with reagent water. Store in a sterilized glass bottle. Refrigerate and prepare monthly.
- Nitrite is easily oxidized, especially in the presence of moisture, and only fresh reagents are to be used.
 - Prepare sterile bottles for storing nitrite solutions by heating for 1 hour at 170°C in an air oven.
- 7.5.4. Bromide stock solution (1.00 mL = 1.00 mg Br^-): Dry approximately 5.0 g of sodium bromide (NaBr) for 12 hours at 105°C , and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.2876 g of the dried salt in reagent water and dilute to volume with reagent water.

- 7.5.5. Nitrate stock solution (1.00 mL = 1.00 mg NO₃⁻ - N): Dry approximately 10.00 g of sodium nitrate (KNO₃) at 105°C for 24 hours . In a 1-liter volumetric flask, dissolve 7.2200 g of the dried salt in reagent water and dilute to volume with reagent water.
- 7.5.6. Phosphate stock solution (1.00 mL = 1.00 mg PO₄⁻ - P): Dry approximately 10.00 g of potassium dihydrogen phosphate (KH₂PO₄) for 1 hour at 105°C and cool in a desiccator. In a 1 liter volumetric flask, dissolve 4.3937 g of the dry salt in reagent water and dilute to volume with reagent water.
- 7.5.7. Sulfate stock solution (1.00 mL = 1.00 mg SO₄⁻²): Dry approximately 5.00 g of potassium sulfate (K₂SO₄) at 105°C for 1 hour and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.8141 g of the dried salt in reagent water and dilute to volume with reagent water.
- 7.6. Working standards: Prepare calibration standard #5 in a 250 mL volumetric flask and transfer to a vial. Adjust the amount of stock solution used to prepare the working standards if the stock concentration differs from 1000 mg/L as assumed below.

Calibration Standard #5

Analyte	mL of Stock	Final Conc.
Fluoride	2.5 mL	10.0 mg/L
Chloride	50 mL	200. mg/L
Nitrite	2.5 mL	10.0 mg/L
Bromide	10 mL	40.0 mg/L
Nitrate	2.5 mL	10.0 mg/L
Ortho-Phosphate	2.5 mL	10.0 mg/L
Sulfate	50 mL	200. mg/L

- 7.6.1. In 5 mL Poly Vials prepare the following calibration standards in reagent grade water. Final concentrations of working standards are shown below.

Calibration Standard #4: take 2.50 mL of calibration standard #5 and add 2.50 mL of reagent water.

Calibration Standard #2: take 250 μ L of calibration standard #5 and add 4.75 mL of reagent water.

Calibration Standard #1: take 25.0 μ L of calibration standard #5 and add 4.95 mL of reagent water.

Calibration Standard #3: take 1.25 mL of calibration standard #5 and add 3.75 mL of reagent water.

Calibration Standard #1

Analyte	25.0 μ L of Cal Std #5	Final Conc
Fluoride		0.05 mg/L
Chloride		1.0 mg/L
Nitrite		0.05 mg/L
Bromide		0.20 mg/L
Nitrate		0.05 mg/L
Ortho-Phosphate		0.05 mg/L
Sulfate		1.0 mg/L

Calibration Standard #2

Analyte	250 μ L of Cal Std #5	Final Conc.
Fluoride		0.5 mg/L
Chloride		10. mg/L
Nitrite		0.5 mg/L
Bromide		2.0 mg/L
Nitrate		0.5 mg/L
Ortho-Phosphate		0.5 mg/L
Sulfate		10. mg/L

Calibration Standard #3

Analyte	1.25 mL of Cal Std #5	Final Conc.
Fluoride		2.5 mg/L
Chloride		50. mg/L
Nitrite		2.5 mg/L
Bromide		10. mg/L
Nitrate		2.5 mg/L
Ortho-Phosphate		2.5 mg/L
Sulfate		50. mg/L

Calibration Standard #4

Analyte	2.5 mL of Cal Std #5	Final Conc.
Fluoride		5.0 mg/L
Chloride		100 mg/L
Nitrite		5.0 mg/L
Bromide		40. mg/L
Nitrate		5.0 mg/L
Ortho-Phosphate		5.0 mg/L
Sulfate		100 mg/L

7.6.2. Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards to 200 mL in a volumetric flask as indicated in the table below to prepare the mixture to be used for the LCS/matrix spike and CCV solution. Adjust the amount of stock solution used to prepare the working standards if the stock concentration differs from 1000 mg/L as assumed below.

LCS / Matrix Spiking & Continuing Calibration Verification Solution

Analyte	mL of 2° Stock Soln	Final Conc. (V _f =200ml)
Fluoride	0.50 mL	2.5 mg/L
Chloride	10. mL	50. mg/L
Nitrite	0.50 mL	2.5 mg/L
Bromide	2.0 mL	10. mg/L
Nitrate	0.50 mL	2.5 mg/L
Ortho Phosphate	0.50 mL	2.5 mg/L
Sulfate	10. mL	50. mg/L

NOTE: Stock standards (7.5), calibration standard #5 (7.6) and LCS standard (7.6.2) should be stored in the dark at 4 °C. Replace these standards when instrument response indicates target analyte degradation may have occurred or after 6 months, whichever occurs first. Nitrite and ortho-phosphate are particularly light and oxygen sensitive.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.
- 8.2. Sample preservation and holding times for the anions that can be determined by this method for water samples are as follows:

Analyte	Preservation	Holding Time
Fluoride	4° C	28 days
Chloride	4° C	28 days
Nitrite	4° C	48 hours
Bromide	4° C	28 days
Nitrate	4° C	48 hours
Ortho Phosphate	4° C	48 hours
Sulfate	4° C	28 days

Note: Soil leachates will follow the same preservation and holding times as the water samples: starting from the time of extraction.

9. QUALITY CONTROL

- 9.1. The Quanterra QC Program document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.
- 9.2. Table I provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.
- 9.3. Initial Demonstration of Capability
 - 9.3.1. Prior to the analysis of any samples by Ion Chromatography, the following requirements must be met:
 - 9.3.1.1. Method Detection Limit (MDL): An MDL must be determined prior to analysis of any samples. The MDL is determined using seven replicates of reagent water spiked with the anions of interest or standard solid reference material spiked with the anions of interest that has been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis. The spike level must be greater than the calculated MDL should be less than 10x the MDL. The result of the MDL determination must be below the Quanterra reporting limit.
- 9.4. Batch definition: Preparation and QC batch definitions are provided in the Quanterra QC Policy.
- 9.5. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of reagent grade water that has been taken through the entire preparation and analytical process. 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 above the reporting limit.
- 9.6. Laboratory Control Sample (LCS): One LCS must be processed with each preparation batch and must be carried through the entire analytical procedure. 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 the result is outside established control limits the system is out of control and corrective action must occur. Until in-house limits are established, a control limit of 90 - 110% recovery must be applied. Corrective action will

be reparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable. The LCS consists of reagent grade water containing a known amount of target analytes that has been injected into the ion chromatography system. The LCS is prepared from a separate stock standard, or neat material, of a different manufacturer than the stock, or neat material, used to prepare the calibration standard.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each QC batch. A matrix spike (MS) is a field sample to which a known concentration of target analyte has 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 DQO's may require the use of sample duplicates in place of or in addition to MS/MSD's. 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. Spiking levels will be the same as the LCS values.

- If the MS/MSD recovery or RPD falls outside the acceptance range, the recovery of the analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 90-110% recovery and 20% RPD must be applied to the MS/MSD.
- 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 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 the limits, corrective action must be taken. Corrective action will include reparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample volume then a LCS duplicate must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike limits.

9.8. Continuing Calibration Verification (CCV/CCB): Continuing calibration is verified by analyzing the calibration standard after every ten (10) samples. The CCV must fall within +/- 10% of the true value for each target analyte. A CCB is analyzed immediately following the CCV to monitor low level accuracy and system cleanliness. The CCB result must be below the reporting limit for that analyte. If either the CCV or CCB fail to meet criteria, the analysis must be terminated, the problem corrected and reparation and analysis of all samples following the last CCV and CCB which were in control.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Establish ion chromatographic operating parameters equivalent to those indicated in table 2.
- 10.2. For each analyte of interest, prepare 5 calibration standards and a blank by adding accurately measured volumes of one or more stock standards to a volumetric flask and dilution to volume with reagent water. If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible then three new calibration concentrations must be chosen, two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.
- 10.3. Using an injection volume of 25 uL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded. All analytes will be calibrated using a quadratic regression forced through the origin. Correlation coefficients (R^2) must be 0.995 or better.

11. PROCEDURE

- 11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- 11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.
- 11.3. Table 2 summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Sect. 9.2 are met.
- 11.4. Check system calibration daily and, if required, recalibrate as described in Sect 10.
- 11.5. Load and inject a fixed amount (25 uL) of settled & filtered sample. If the sample is cloudy then it should be filtered prior to loading into the autosampler polyvial. Flush injection loop thoroughly. using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.
- 11.6. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of various concentration. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms since retention time is concentration dependent for most analytes..
- 11.7. If the response for the peak exceeds the working range of the system. dilute the sample with an appropriate amount of reagent water and reanalyze.

-
- 11.8. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

- 11.9. The following extraction should be used for solid materials: Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This slurry is mixed for ten minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45 u membrane type filter. This can be the type that attaches directly to the end of the syringe. Care should be taken to show that good recovery and identification of peaks is obtained with the user's matrix through the use of fortified samples.
- 11.10. Should more complete resolution be needed between peaks the eluent (7.3) can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

12. DATA ANALYSIS AND CALCULATIONS

- 12.1. Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.
- 12.2. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.
- 12.3. Report results in mg/L for aqueous samples and mg/Kg for solid sample leachates.
- 12.4. Report NO_2^- as N
 NO_3^- as N
 HPO_4^- as P

13. METHOD PERFORMANCE

- 13.1. The reporting limits for the following analytes are based on a 25 uL injection volume:

Analyte	Water RL	Soil RL
Fluoride	1.0 mg/L	10 mg/kg
Chloride	1.0 mg/L	10 mg/Kg
Nitrite	0.5 mg/L	5 mg/kg
Bromide	0.5 mg/L	5 mg/Kg
Nitrate	0.05 mg/L	0.5 mg/Kg
O-Phosphate	0.5 mg/L	5 mg/kg
Sulfate	1.0 mg/L	10 mg/Kg

13.2. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in it's use and has the required experience. The analyst must be given two blind performance samples to analyze or process for analysis. Upon successful completion of the performance evaluation (PE) samples, these analyses will be documented as initial qualification,. Requalification must be performed annually thereafter for this procedure. The group/team leader must document the training and PE performance and submit the results to the QA Manager for inclusion in the associate's training files.

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 this procedure must be segregated, and disposed of according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Method 300.0. "Determination of Inorganic Anions by Ion Chromatography". Environmental Monitoring Systems Laboratory, Office of Research and Development. U.S. Environmental Protection Agency, Cincinnati, Ohio. Revision 2.1. August 1993.

16.2. Method 9056, "Determination of Inorganic Anions by Ion Chromatography", SW846,
Test Methods for Evaluating Solid Waste, Third Edition, Update II, September 1994.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

- 17.1. Attachment #1, method Flow Chart
- 17.2. Table 1, Quality Control Samples
- 17.3. Table 2, Standard Instrument Operating Parameters
- 17.4. Figure 1, Example Chromatogram

Determination Of Inorganic Anions By Ion Chromatography

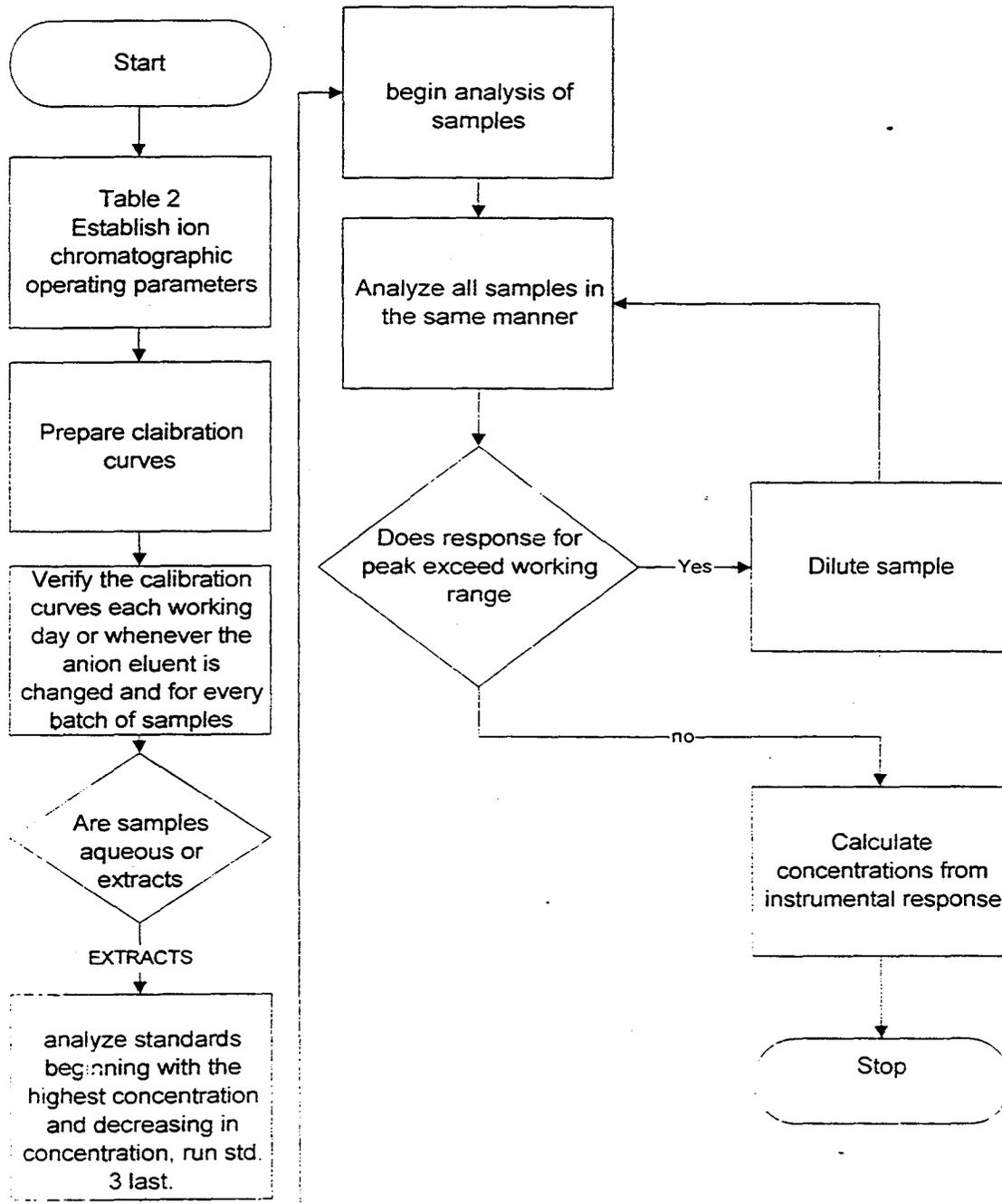


TABLE 1
QUALITY CONTROL SAMPLES

QC Samples	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	At the start of each day following calibrating prior to sample analysis	+/- 10% of true value	Recalibrate and reanalyze
Initial Calibration Blank (ICB)	After Initial Calibration Verification and prior to sample analysis	< the Reporting Limit	Reprepare and reanalyze
Laboratory Control Sample (LCS)	1 per batch of 20 samples	Meets laboratory historical limits	Reanalyze all samples associated with unacceptable LCS
Matrix Spike Sample (MS/MSD)	1 MS/MSD pair per batch or 20 samples	Meets laboratory historical limits	Supervisor's technical judgment
Continuing Calibration Verification (CCV)	Between each group of 10 samples and at the end of the analytical sequence	+/- 10% of true value	Recalibrate and reanalyze all samples since the last acceptable CCV
Continuing Calibration Blank (CCB)	Between each group of 10 samples and at the end of the analytical sequence	< the Reporting Limit	Recalibrate and reanalyze all samples since the last acceptable CCB

TABLE 2
Standard Instrument Operating Parameters

Standard Conditions:

Eluent Pump Rate: 2.0 mL/min.
 Regenerant Flow Rate : 4 mL/min.
 Sample Loop: 25 uL
 Eluent: 1.7mM sodium bicarbonate, 1.8mM sodium carbonate
 Regenerant : 0.025N H₂SO₄.
 Detector output Baseline conductivity should be 15 - 20 uS prior to sample analysis.

Retention Time Matrix (minutes)*

Analyte	Concentration (mg/L)												
	0.05	0.2	0.5	1	2	2.5	5	10	20	40	50	100	200
F ⁻	1.05	1.05			1.05	1.04	1.03						
Cl ⁻			1.71				1.75			1.83	1.88	1.97	
NO ₂ ⁻	2.09	2.1			2.11	2.14	2.19						
Br ⁻		3.28		3.27			3.23	3.2	3.17				
NO ₃ ⁻	3.81	3.78			3.68	3.62	3.54						
o-PO ₄ ²⁻	6.24	6.23			6.19	6.15	6.07						
SO ₄ ²⁻			8.19				8.12			7.87	7.69	7.43	

* Analyte retention time is concentration dependent for most anions. Retention time increases with increasing concentration for chloride and nitrite. Retention time decreases with increasing concentration for bromide, nitrate, ortho-phosphate and sulfate.

EXAMPLE ION CHROMATOGRAPH

Implementation
date: 02/09/99
BKK 02/09/99

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: CYANIDE PREPARATION METHOD

(SUPERSEDES: REVISION 7, DATED 03/19/98)

Prepared by:	<u>Mark Z. Bunn</u>	<u>2/5/99</u>
		Date
Reviewed by:	<u>Peter O'Meara</u>	<u>2/5/99</u>
	Technology Specialist	Date
Approved by:	<u>Paul M. Cahill</u>	<u>2-5-99</u>
	Quality Assurance Manager	Date
Approved by:	<u>Steve J. ...</u>	<u>2-8-99</u>
	Environmental Health and Safety Coordinator	Date
Approved by:	<u>Christoph R. Opandi</u>	<u>2-5-99</u>
	Laboratory Director	Date

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

1. SCOPE AND APPLICATION.....	3
2. SUMMARY OF METHOD.....	3
3. DEFINITIONS	3
4. INTERFERENCES	3
5. SAFETY	3
6. EQUIPMENT AND SUPPLIES	4
7. REAGENTS AND STANDARDS.....	5
8. SAMPLE PRESERVATION AND STORAGE	9
9. QUALITY CONTROL	9
10. CALIBRATION AND STANDARDIZATION	11
11. PROCEDURE	12
12. DATA ANALYSIS AND CALCULATIONS.....	16
13. METHOD PERFORMANCE.....	17
14. POLLUTION PREVENTION.....	17
15. WASTE MANAGEMENT.....	17
16. REFERENCES.....	17
17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)	18

1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of Total, Amenable, and Free Cyanide in solids, liquids, and waters. It is based on CLP ILM03.3, CLP ILM04.0, SW 846 Method 9012A, EPA 335.1 and 335.2, Standard Method 4500-CN-I and ASTM D 4282-83. The working linear range is 0.005 - 0.1 mg/L for waters and 0.25 to 5 mg/kg for solids.
- 1.2. The associated method codes are WH, N4, CF, HF, DV, OU, CG, VL, A4, VQ, and QP
- 1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

- 2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.
- 2.2. The microdiffusion method (D 4282), releases HCN from the sample by neutralizing the sample with a potassium phosphate solution and trapping the diffused gas in a sodium hydroxide solution.

3. DEFINITIONS

- 3.1. Refer to the glossary in 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 that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2. Interferences for the microdiffusion method (D 4282) include the decomposition of hexacyanoferrates. Precipitation of the hexacyanoferrates with cadmium and diffusion in the dark virtually eliminates this decomposition.

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 (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. The following specific hazards are known:
- 5.3.1. The following are known to be **corrosive: sulfamic acid, sodium hydroxide, sulfuric acid, acetic acid.**
- 5.3.2. Cyanide is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and clean-up techniques before using this material.
- 5.4. 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.
- 5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. Cyanide Distillation Apparatus
- 6.2. Analytical balance. capable of accurately weighing ± 0.0001 g
- 6.3. Vacuum pump
- 6.4. Graduated cylinders: various
- 6.5. Volumetric flasks: various
- 6.6. Volumetric pipets: various
- 6.7. Balance: Top loading. capable of accurately weighing ± 0.01 g

- 6.8. Lead Acetate Indicator Paper
- 6.9. Potassium Iodide (KI) Indicator Paper
- 6.10. Erlenmeyer flasks: various
- 6.11. Buret: Class A 10 mL
- 6.12. pH strips
- 6.13. Boiling stones or chips
- 6.14. Beakers: various
- 6.15. Snap seal containers: 120 mL
- 6.16. Plastic bottles with lids: 250mL or 500mL
- 6.17. Conway microdiffusion cell (83 mm OD, 4 mm moat): Bel Art F40941-0000 or equivalent

7. REAGENTS AND STANDARDS

7.1. Reagents

- 7.1.1. Sulfamic Acid: reagent grade (not used for CLP ILM03.0)
- 7.1.2. Sulfamic Acid Solution: Add 100 g of sulfamic acid to 800 mL reagent water and dilute to 1 liter with reagent water (not used for CLP ILM03.0)
- 7.1.3. Ascorbic Acid: reagent grade
- 7.1.4. Sodium Hydroxide: (NaOH), high purity grade.
- 7.1.5. Sodium Hydroxide, 1.25 N: Add 50 g of NaOH to 900 mL and dilute to 1 liter with reagent water.
- 7.1.6. Sodium Hydroxide, 0.25 N: Add 10 g of to 900 mL reagent water and dilute to 1 liter with reagent water.
- 7.1.7. Sulfuric Acid: (H_2SO_4), concentrated
- 7.1.8. Acetic Acid Solution: Add 10 mL of glacial acetic acid to 90 mL of reagent water.
- 7.1.9. Magnesium Chloride: ($MgCl_2 \cdot 6H_2O$), reagent grade

- 7.1.10. Magnesium Chloride Solution: Add 510 g $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ to 500 mL reagent water and dilute to 1 liter with reagent water.
- 7.1.11. Calcium Hypochlorite: $[\text{Ca}(\text{OCl})_2]$, reagent grade
- 7.1.12. Calcium Hypochlorite Solution: Add 5 g of $\text{Ca}(\text{OCl})_2$ to 100 mL of reagent water.
- 7.1.13. Methyl Red Indicator: Add 0.05 g of methyl red to 50 mL of glacial acetic acid and dilute to 100 mL with reagent water.
- 7.1.14. Methyl red reagent grade glacial
- 7.1.15. Acetic Acid: (CH_3COOH) , glacial reagent grade
- 7.1.16. Zinc Acetate: $[\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)]$, reagent grade
- 7.1.17. Zinc Acetate Solution: Add 100 g zinc acetate to 800 mL reagent water and dilute to 1 liter with reagent water.
- 7.1.18. Sodium Acetate: $[\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}]$ reagent grade
- 7.1.19. Sodium Acetate Buffer: Add 410 g of sodium acetate to 500 mL of reagent water. Adjust the pH to 4.5 using glacial acetic acid and dilute to 1 liter with reagent water.
- 7.1.20. Rhodanine: reagent grade
- 7.1.21. 0.0192 N Silver Nitrate: reagent grade
- 7.1.22. Bismuth nitrate $[\text{Bi}(\text{NO}_3)_3]$: Dissolve 30 g of $\text{Bi}(\text{NO}_3)_3$ in 100 mL of reagent water. While stirring, add 250 mL of glacial acetic acid. Stir until dissolved. Dilute to 1 L with reagent water.
- 7.1.23. Cadmium chloride $[\text{CdCl}_2]$ – anhydrous : Dissolve 1.0 g CdCl_2 in 90 mL of reagent water. Dilute to 100 mL with reagent water to produce a 10 g/L CdCl_2 solution.
- 7.1.24. Potassium Phosphate solution : Dissolve 7 g of NaOH in approximately 200 mL of reagent water in a 1 L beaker. Add 95 g of potassium phosphate monobasic $[\text{KH}_2\text{PO}_4]$ and dilute to 475 mL with reagent water. Once the KH_2PO_4 is dissolved adjust the pH to 6.0 ± 0.1 with 100 g/L NaOH solution. Dilute to 500 mL with reagent water.
- 7.1.25. Standard diluent (0.05 N NaOH) : Dissolve 1.02 g NaOH in 400 mL of reagent water. dilute to 500 mL with reagent water.

7.1.26. Absorber solution (0.1 N NaOH) : Dissolve 2.05 g NaOH in 400 mL of reagent water, dilute to 500 mL with reagent water.

7.2. Standards

7.2.1. Secondary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium ferricyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well. Prepare fresh every 1 - 3 months.

7.2.2. Note: Cyanide stock standard must be standardized prior to use (See Section 0) Working Standards: Add the required amounts of 1.25 N NaOH and cyanide standards to volumetric flasks according to the suggested chart. Dilute to volume using reagent water. See chart below:

Working Standards					
Volume 1.25 N NaOH (mL)	Std Volume (mL)	Std Conc. (mg/L)	Final Volume (mL)	Final Conc. (mg/L)	Expiration Time (Days)
20	10	1000	100	100	14
20	10	100	100	10	7
20	10	10	100	1	1

7.2.3. Working Standards for microdiffusion option : Prepare 1.0 mg/L primary and secondary standards by diluting 0.10 mL of the corresponding stock standards (1000 mg/L) to 100 mL with the standard diluent (0). Store in an opaque container for a maximum of 7 days.

7.2.4. Prepare the initial calibration standards, ICV and at least two CCVs according to this table for the micro diffusion preparation.

Working Std	L of Std	Standard Diluent (0.05 N NaOH)	Conc (mg/L)
1°	0.30	2.70	0.10
1°	0.15	2.85	0.050
1°	0.075	2.90	0.025
1°	0.030	2.95	0.010
1°	0.015	3.00	0.005
2°	0.075	2.90	0.025 (ICV)
1°	0.075	2.90	0.025 (CCV)

7.2.5. Prepare a base & gain standard (0.23 mg/L) for the autoanalyzer. This is the same concentration as the 0.10 mg/L standard after it has undergone the 2.3X concentration effect in the microdiffusion preparation. Put 1.15 mL of 1° working standard (0) in a 5 mL autoanalyzer cup and add 3.85 mL of reagent water.

7.2.6. Cyanide Standardization

7.2.6.1. Using a volumetric pipet, aliquot 10.0 mL of the 1000 ppm stock cyanide standard into an Erlenmeyer flask and add 90 mL of reagent water.

7.2.6.2. Add 10 drops of Rhodanine indicator.

7.2.6.3. Titrate with 0.0192 N Silver nitrate until the color changes from yellow to pink/orange.

7.2.6.4. Calculation:

$$\text{Cyanide, mg/L} = \frac{(A) (1000)}{\text{mL cyanide solution (10)}}$$

Where: A = mL Titrant for standard

7.2.6.5. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

8. SAMPLE PRESERVATION AND STORAGE

- 8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH>12. All samples are stored at 4° C ± 2°C in plastic or glass containers.
- 8.2. The holding time is fourteen days from sampling to analysis.

NOTE: CLP samples holding time is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

- 9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, MS, MSD, Method Blanks) which are processed similarly, with respect to the procedure. All samples within the batch must be treated with the same lots of reagents and the same processes. All samples within the batch must be treated with the same processes.

9.2. Method Blank (MB)

- 9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.
- 9.2.2. A blank consisting of 50 mL reagent water must be prepared and distilled (or diffused, 3 mL) with each analytical batch of samples.
- 9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. 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.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. 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.

9.3.2. A midrange LCS consisting of 2.0 mL of the 1.0 mg/L standard (secondary source) brought to a final volume of 50 mL must be distilled with each analytical batch of samples.

9.3.3. The microdiffusion LCS is prepared by taking 0.12 mL of 2° working standard (0) and adding 2.85 mL of standard diluent (0).

9.3.4. Corrective Action for Laboratory Control Samples

9.3.4.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.4.2. Corrective action will be reparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each 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/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only

on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

- 9.4.2. A MS/MSD consisting of 50 mL or 1.0 g of sample brought up to 50 mL with reagent water and 2.0 mL of the 1.0 mg/L standard (either source) must be distilled with each analytical batch of samples.
- 9.4.3. Prepare the microdiffusion MS/MSD by adding 0.12 mL of 2° working standard (0) to 3.0 mL of sample for each batch of samples.
- 9.4.4. Corrective action for MS/MSDs
- 9.4.4.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.
- 9.4.4.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data reported as DIL (diluted out).
- 9.4.4.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

NOTE: MS for CLP samples require 5 mL of 1.0 mg/L standard (true 100 µg/L)

- 9.5. Sample Duplicates are required for all microdiffusion prepared samples

10. CALIBRATION AND STANDARDIZATION

- 10.1. For all non-CLP methods, a low and high standard are distilled from the same source as the calibration curve each day. Prepare the low standard (0.025 mg/L) by diluting 0.125 mL of 10.0 mg/L standard with reagent water to a final volume of 50 mL. Prepare the high standard (0.075 mg/L) by diluting 0.375 mL of a 10.0 mg/L standard with reagent water to a final volume of 50mL. Method 9012A *recommends* that the distilled standards (a high and a low) be compared to the curve with a ±10% agreement with the undistilled standards. The distilled standards are evaluated against all applicable batch QC.
- 10.2. All standards (initial calibration, ICV, CCV) for the microdiffusion preparation option must be processed through the diffusion prep at the same time and in the same manner as the samples.

11. PROCEDURE

11.1. Summary

11.1.1. For all non-CLP methods, the sample is distilled/refluxed under acidic conditions for one hour. For CLP methods, the samples are distilled for one and a half hours. The released HCN is trapped in 0.25 N NaOH solution.

11.1.2. The microdiffusion method (D 4282), releases HCN from the sample by neutralizing the sample with a potassium phosphate solution and trapping the diffused gas in a sodium hydroxide solution

11.2. Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.2.1. Checking for Interferences (For non-CLP samples)

11.2.1.1. Using pH paper strips, check the pH of the sample and record it as >12 or <12 on the analytical logsheet. If pH is <12, the deviation **must be addressed in the project narrative.**

11.2.1.2. Using lead acetate paper, check the sample for excess sulfide. If sulfide is present the test strip will turn silver/brown. Record findings on the analytical logsheet. If sample contains sulfide, treat the sample by adding 5 mL of bismuth nitrate solution. Mix for 3 minutes prior to addition of H₂SO₄. The calibration curve must be distilled in the same manner as the samples. Prepare a curve as follows:

mL of 1.0 mg/L		Final Concentration
<u>CN Standard</u>	<u>Final Volume (mL)</u>	<u>(mg/L CN)</u>
0.25	50	0.005
0.5	50	0.010
1.25	50	0.025
2.50	50	0.050
5.0	50	0.100

Using potassium iodide paper, check for chlorine presence. If chlorine is present the test strip will turn dark blue. If chlorine is present add 0.1 - 0.5 grams ascorbic acid to sample until the chlorine is destroyed. Record this on the analytical logsheet.

11.2.1.3. For CLP samples: Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material.

11.2.2. Complex Cyanide Preparation Procedure [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]: Add 100 mL of water sample or 2.0 g of solid sample and 100 mL reagent water to a plastic, sealable container. Add 10 g NaOH pellets. Mix or tumble for 12-16 hours. Allow solids to settle out. Transfer 50 mL of the solution to the distillation tube. Distill sample (See section 11.2.4.1).

Note: If the sample requires an MS/MSD or MS/Dup, double or triple the volumes as stated above. A blank and LCS should be prepared and analyzed with samples using 10 g NaOH and 100 mL reagent water.

11.2.3. Amenable Cyanide (Chlorinated Aliquot) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.2.3.1. Place 50 mL (waters) or 1.0 g (solids/liquids) into a beaker. Add 50 mL of reagent water to non-waters. Place the beaker on a stir plate and begin mixing. Test the pH of the solution, if less than 12 add 1.25 N NaOH, drop by drop until pH \geq 12. Drop by drop, add calcium hypochlorite until an excess of chlorine is reached. Test for chlorine excess using KI paper. Allow the sample to chlorinate for one hour. At the end of the chlorination period, add about 0.1 to 0.5 g of ascorbic acid to destroy excess chlorine. Test using KI paper. Keep the addition of ascorbic acid to a minimum. Pour the sample into a distillation flask and follow the total cyanide preparation method (Section 11.2.4). Also set up an unchlorinated aliquot of sample (50 mL or 1.0 g) following the total cyanide method.

11.2.4. Total Cyanide

11.2.4.1. Add 50 mL or 1.0 g of the sample and 2 - 3 boiling chips to the distillation tube. Bring all volumes to 50 mL with reagent water. Add 50 mL of

0.25 N NaOH solution to the absorber tube and assemble the scrubber.
Assemble the cyanide distillation apparatus.

11.2.4.2. Turn on the cooling water to "60". Turn on the vacuum source and adjust the flow such that even stream of air bubbles are in the scrubber tube approximately $\frac{1}{4}$ " of foam or 1-2 bubbles per second. At this time add any spiking solutions to the LCS or MS/MSDs samples directly into the inlet tube.

11.2.4.3. In the order stated, add 2.0 mL sulfamic acid solution (not for CLP ILM03.0 samples), 2.5 mL of concentrated sulfuric acid and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions.

Note: Complex cyanide samples require an additional 2.5 mL of sulfuric acid. Be sure the pH is < 2 .

11.2.4.4. Flip the heater switch to "on" and turn the dial to an appropriate setting (according to manufacturer's instructions) to allow the apparatus to warm up. Be sure to adjust the air flow and water as necessary. Heat for one hour. After the heating period, the heater will turn off automatically. Allow to cool for fifteen minutes. Keep the vacuum and cooling water on.

NOTE: For CLP, heat samples for one and a half hours.

11.2.4.5. Disconnect the absorber. Pour solution into a 120 mL snap seal container. Do not rinse the scrubber tube or dilute NaOH in the snap seal. Be sure to properly label the bottles "total" or "amenable" along with sample ID, position and date.

11.2.5. Free Cyanide (Weak and Dissociable) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.2.5.1. Add 50 mL or 1.0 g of sample to the distillation tube. Bring all volumes up to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH to the absorber tube and assemble.

11.2.5.2. Turn on cooling water to "60". Turn on the vacuum source. Add any spiking standards to the appropriate LCS or MS/MSDs at this time. Through the inlet tube, add 1.0 mL of sodium acetate buffer, 1.0 mL of zinc acetate, and 0.25 mL of methyl red indicator. Rinse the inlet tube sparingly with reagent water between and after reagent additions. If the sample is not red, carefully add 1:9 acetic acid dropwise until the sample does turn red.

11.2.5.3. Flip on the heater switch and turn the dial to "10.5" (This allows the apparatus to warm up). Adjust the air flow and water as needed. Heat for 1 hour. After the heating period, the heater will shut off automatically. Allow the sample to cool for fifteen minutes with the air and water on.

11.2.5.4. Pour the scrubber contents into a 120 mL snap seal bottle. Do not rinse the scrubber. Label the bottle well and be sure to denote that it is a "free" sample distillate.

11.2.6. Cyanide Unit Clean-up

11.2.6.1. Cyanide distillation unit glassware is very fragile and expensive. It must be handled with care at all times.

11.2.6.2. Disassemble each set-up and rinse the sample down the drain with large amounts of water. Be sure to collect the solids in a screen and dispose of properly.

11.2.6.3. Wash each set-up with soap and hot water, and then rinse with 1:1 HCl. Rinse several times with reagent water

11.2.6.4. Re-assemble the set-up.

11.2.6.5. Be sure to wash each set up as a separate unit and replace in the same position.

11.2.6.6. If a sample of known high cyanide concentration was distilled in a certain position, be sure to change the appropriate tubing on that position.

11.3. Microdiffusion Sample Preparation Procedure

11.3.1. Pipet 3.0 mL of sample (or appropriately diluted standard [0.0]) into the middle ring of a clean and dry Conway diffusion cell. The outer ring is used as part of the lid sealing mechanism. Cover the cell.

11.3.2. Lift the cover and pipet 1.3 mL of absorber solution (0.1 N NaOH [0]) into the center chamber of the diffusion cell. Recover the cell.

11.3.3. Lift the cover and pipet 0.5 mL of cadmium chloride solution [0] into an open area of the sample ring (i.e. middle ring) of the cell. Recover the cell. When all cells have been treated with cadmium chloride, lift the tray holding all cells and

gently tilt back and forth for 30 seconds to mix. Caution do NOT tilt so far that the absorber solution or sample go over the short wall that separates them.

11.3.4. Lift the cover of each cell individually and pipet 1.0 mL of potassium phosphate solution [0] into an open area of the sample ring. Cover immediately. . When all cells have been neutralized, lift the tray holding all cells and gently tilt back and forth for 30 seconds to mix. Caution do NOT tilt so far that the absorber solution or sample go over the short wall that separates them.

11.3.5. Carefully place the tray containing all the diffusion cells in darkened drawer for 4 to 8 hours. Do NOT tip the tray so far that the absorber and sample solutions can mix.

11.3.6. After the diffusion time is complete, carefully remove the tray / diffusion cells from the drawer. Using a disposable plastic pipet transfer the absorber solution from the cell chamber of the cell to a 5 mL autoanalyzer cup. If the absorber solution will not be immediately analyzed place a 2 mL autoanalyzer cup on top as a cap.

11.3.7. Wash each cell and cover with soap and tap water. Do not scrub with a brush or "scratch" pad unless necessary to remove material adhering to the inside of the cell. Rinse well with tap and deionized water. Place on the tray to dry.

11.4. Analytical Documentation

11.4.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.4.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.4.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.4.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. 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 experience.

14. POLLUTION PREVENTION

14.1. Using a Midi Cyanide distillation unit saves time and requires less sample and reagents for use.

15. WASTE MANAGEMENT

15.1. Acid waste must be collected in clearly labeled acid waste cans.

15.2. Solid waste materials (soil, gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Refer to the Laboratory Sample and Waste Disposal plan

16. REFERENCES

16.1. References.

16.1.1. SW846. Test Methods for Evaluating Solid Waste, Third Edition, Total and Amenable Cyanide. Automated UV: Method 9012A.

16.1.2. EPA 600: Cyanide. Total and Cyanide, Amenable to Chlorination: Methods 335.1 and 335.2

16.1.3. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition: Weak and Dissociable Cyanide: Method 4500-CN-I

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition: Complex Cyanide: Method 4500-CN-E

16.1.5. USEPA CLP SOW ILM03.0 Section D-Cyanide Midi Distillation

16.1.6. Quanterra[®] Quality Assurance Management Plan, current revision

16.1.7. ASTM Standard D 4282-83, Determination of Free Cyanide in Water and Wastewater by Microdiffusion.

16.2. Associated SOPs

16.2.1. Cyanide, Automated Method NC-WC-0031

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. BASF RFI Requirements

17.1.1. Amendments to this SOP to satisfy BASF RCRA Facility Investigation (RFI) requirements are based on comments from US EPA upon review of site requirements and laboratory procedures.

17.1.2. Amendment to the preparation of **all** samples

17.1.2.1. **Section 7.2.1:** Change 2.51 g of potassium cyanide to 2.11g potassium ferricyanide.

17.1.2.2. **Section 9.4:** The matrix spike/duplicate frequency has been increased from one per batch to one per 7 samples. Consult with the project manger for specific samples to be spiked. The goal is to spike each sampling area at the site at least once.

17.1.3. Amendment to the preparation of **Prussian blue area** samples

17.1.3.1. **Section 7.1.22:** Sodium thiocyanate (reagent grade): Prepare a spiking solution at a concentration 100X greater than the measured cyanide for the appropriate Prussian blue area samples.

17.1.3.2. **Section 9.5:** Three Prussian blue area samples will be spiked with 1 mL of the sodium thiocyanate solution (Section 17.1.3.1) in order to assess the potential impact of thiocyanate in the original samples on the measured cyanide concentration. These matrix spikes do not replace the cyanide matrix spikes mentioned above (Section 17.1.2.2). The increase (if any) in measured cyanide concentration relative to the original sample shall be reported as “percentage increase” using the following equation:

$$\%CN_{increase} = \frac{Conc_{SCN_spike} - Conc_{unspiked}}{Conc_{unspiked}} \times 100$$

where

$Conc_{SCN_spike}$ = cyanide concentration measured in the thiocyanate spiked sample

$Conc_{unspiked}$ = cyanide concentration measured in the unspiked sample

Note: Section 11.3.2.1 of NC-WC-0031 addresses sulfide testing and precipitation.

17.2. Method Deviation (9012A. 335.1. 335.2)

17.2.1. The reflux distillation apparatus used is the midi distillation.

17.2.2. The volume of sample used is reduced to 50 mL vs. 500 mL using the midi-distillation apparatus.

17.2.3. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

17.2.4. The analysis of samples prepared by microdiffusion uses the concentrations and volumes specified in SOP NC-WC-0031 rather than the reagent concentrations and volumes specified in ASTM D 4282-83.

Implementation Date: _____

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

(SUPERSEDES: REVISION 3, DATED 03/16/98)

Prepared by: Patrick O'Meara 2-16-99
Date

Reviewed by: Laurel E. Wood 2-16-99
Technology Specialist Date

Approved by: Qualita Fob 2-11-99
Quality Assurance Manager Date

Approved by: Steve J... 2-17-99
Environmental Health and Safety Coordinator Date

Approved by: Anthony J. Operti 2-18-99
Laboratory Director Date

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend, or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD	3
3. DEFINITIONS	3
4. INTERFERENCES	3
5. SAFETY	3
6. EQUIPMENT AND SUPPLIES	4
7. REAGENTS AND STANDARDS	5
8. SAMPLE COLLECTION, PRESENTATION AND STORAGE.....	7
9. QUALITY CONTROL	7
10. CALIBRATION AND STANDARDIZATION.....	9
11. PROCEDURE.....	10
12. DATA ANALYSIS AND CALCULATIONS.....	13
13. METHOD PERFORMANCE	14
14. POLLUTION PREVENTION.....	15
15. WASTE MANAGEMENT	15
16. REFERENCES.....	15
17. MISCELLANEOUS (TABLES, APPENDICES, ETC...).....	16

1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of Total, Amenable, and Free Inorganic Cyanide in solids, liquids, and waters. It is based on CLP ILM03.0, SW846 Method 9012A, EPA 335.1 and 335.2, and Standard Method 4500-CN-I. The working linear range is 0.005 - 0.1 mg/L for waters and 0.25 to 5 mg/kg for solids.
- 1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

- 2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.
- 2.2. The sodium hydroxide solution is analyzed colorimetrically on an autoanalyzer using the pyridine-barbituric acid method.

3. DEFINITIONS

- 3.1. Refer to the glossary in 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 that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2. Sulfides interfere, but can be eliminated by treating sodium hydroxide distillate with cadmium carbonate prior to analysis.

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. The following specific hazards are known:
 - 5.3.1. The following materials are known to be **corrosive**: **Sodium Hydroxide**
 - 5.3.2. Cyanide is a highly toxic compound that must be handled with care. The analyst must be aware of the handling techniques.
- 5.4. 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.
- 5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. Traacs 800 autoanalyzer
- 6.2. Probe 0.016"
- 6.3. Flowcell 10nm
- 6.4. Wavelength cell 570nm
- 6.5. Manifold-Multitest cartridge with cyanide tubing
- 6.6. 125 mL Erlenmeyer flasks

- 6.7. 100 mL, 250 mL, 1000 mL volumetric flasks
- 6.8. Volumetric pipets: various
- 6.9. Top loading balance: capable of accurately weighing ± 0.01 g.

7. REAGENTS AND STANDARDS

7.1. Reagents

- 7.1.1. Cadmium carbonate: powder
- 7.1.2. 1.25 N sodium hydroxide: Add 50 g of sodium hydroxide pellets (NaOH) to a 1 liter volumetric flask and dilute to volume with reagent water.
- 7.1.3. Phosphate buffer: Add 136 g of potassium phosphate - monobasic (KH_2PO_4) and 2.8 g of sodium phosphate - dibasic (Na_2HPO_4) to 800 mL of reagent water in a 1 liter volumetric flask. Mix, bring to volume with reagent water. Add 3-5 drops of Brij-35 to 100 mL of Buffer prior to using.
- 7.1.4. Chloramine-T reagent: Add 1.0 g of chloramine-T to a 250 mL volumetric flask and dilute to volume with reagent water. Prepare fresh daily.
- 7.1.5. Pyridine reagent: Add 15.0 g of barbituric acid to a 1 liter volumetric flask. Add 75 mL of pyridine and 15 mL of concentrated hydrochloric acid (HCl) and mix. Bring to volume with reagent water and store at $4^\circ\text{C} \pm 2^\circ\text{C}$ in an amber glass bottle.
- 7.1.6. 0.25 N sodium hydroxide: Add 200 mL of N NaOH to a 1 liter volumetric flask and dilute to volume with reagent water.
- 7.1.7. Brij-35
- 7.1.8. 30% Brij solution: Add 2 mL Brij-35 to 1000 mL reagent water.
- 7.1.9. Rhodanine indicator, purchased.
- 7.1.10. 0.0192 N silver nitrate, purchased

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months.

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1. Pipet the appropriate amount of cyanide standard into 100 mL volumetric. Add 20 mL 1.25N NaOH to each calibration standard (except the 1.0 mg/L) and bring to volume with reagent water. Prepare weekly.

<u>Concentration CN-</u>	<u>mL CN-</u>	<u>Final Volume</u>
100 mg/L	10 mL of 1000 mg/L	100 mL
10 mg/L	10 mL of 100 mg/L	100 mL
1.0 mg/L	10 mL of 10 mg/L	100 mL
*0.1 mg/L	10 mL of 1 mg/L	100 mL
*0.05 mg/L	5 mL of 1 mg/L	100 mL
*0.025 mg/L	25 mL of 0.1 mg/L	100 mL
*0.01 mg/L	10 mL of 0.1 mg/L	100 mL
*0.005 mg/L	10 mL of 0.05 mg/L	100 mL

*Denotes calibration standards

NOTE: The 1 ppm CN standard is only good for the day it is prepared and is not preserved with NaOH.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH > 12. All samples are stored at 4°C ± 2°C in plastic or glass containers.
- 8.2. The holding time is fourteen days from sampling to analysis.

*NOTE: Holding time for CLP is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

- 9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

- 9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.
- 9.2.2. A reagent water blank consisting of 50 mL reagent water must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-0032 for distillation instructions.
- 9.2.3. Corrective Action for Blanks
 - 9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. 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.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. 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.

9.3.2. A midrange LCS consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L to 50 mL) must be distilled and analyzed with each analytical batch of samples. See Method LM-WALN-1142 (NC-WC-0032) for distillation instruction.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be re-preparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each 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/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. A MS/MSD consisting of 50 mL or 1.0 g sample and 0.04 mg/L spike (2.0 mL of 1.0 mg/L to 50 mL) will be distilled and analyzed with every batch. See SOP NC-WC-0032 for distillation instructions..

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include reparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as DIL (diluted out).

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. The instrument is calibrated using five cyanide standards at the time of analysis.

10.2. Initial Calibration

10.2.1. The instrument is calibrated at the beginning of each run and is verified at the beginning of the run by using a midrange ICV. The correlation coefficient of the original curve must be ≥ 0.995 or recalibration is required.

10.3. Continuing Calibration

10.3.1. The run is checked every ten samples and at the end of the run using a midrange CCV to verify continued linearity. It cannot vary from the original curve by more than $\pm 15\%$ or recalibration is required. The CCV is composed of the 0.025 ppm secondary standard.

10.3.2. System cleanliness is checked every ten samples and at the end of the run using a CCB. It cannot contain the analyte of interest above the reporting limit or recalibration is required. The CCB is 0.25N NaOH.

NOTE: Base and gain values may change with new reagents, standards, tubing changes, or board cleaning. These values are for reference only.

10.4. High and Low Standard

10.4.1. The distillation technique is checked by distilling a high and low standard and comparing the values obtained to the standard curve. If the percent recovery is not within $\pm 10\%$, the analyst will submit a Nonconformance Memo for the analytical run.

10.5. Normal Tray Protocol

10.5.1. P,5C>2, H@2, 2L@0, G@2, XS>7, H@2, 2L@0, G@2, E

NOTE: X = number of sample cups minus the six calibration cups.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. See cyanide distillation SOP: NC-WC-0032.

11.3.1.1. The sample is distilled/refluxed under acidic conditions for one hour. The released HCN is trapped in 50 mL of 0.25 N NaOH solution.

NOTE: CLP samples are distilled for one and a half hours.

11.3.2. Sample Preparation Procedure

11.3.2.1. Non-CLP Samples: For samples containing sulfide, calibrate the instrument using the calibration curve specially prepared for samples containing sulfide.

11.3.2.2. CLP Samples: Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material

11.4. Sample Analysis

11.4.1. Summary

11.4.1.1. The sample distillates are analyzed on the autoanalyzer for cyanide using the automated pyridine-barbituric method.

11.4.2. Recommended Instrument Conditions

11.4.2.1. See Manufacturer's information for operation instructions.

11.4.2.2. Start Up Solutions (and Wash Solutions)

11.4.2.2.1. All lines in 0.25N NaOH solution:

11.4.2.2.2. Then all lines in 30% Brij solution.

11.4.2.3. Running Solutions

11.4.2.3.1. Two DI water lines (orange/green) in 30% Brij solution

11.4.2.3.2. Red/Red in pyridine reagent

11.4.2.3.3. Orange/red in chloramine-T reagent

11.4.2.3.4. White/white in phosphate buffer solution

11.4.2.4. Base and Gain

11.4.2.4.1. Performed on the 0.1 mg/L standard

11.4.2.4.2. Approximate values: Base = 75 Gain = 150

*These values are for reference only and may change with new reagent or standard preparations.

11.4.2.5. Trouble shooting for poor Base and Gain or baseline noise

11.4.2.5.1. Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes.

Place lines back in reagents and perform another base and gain when the baseline has stabilized.

11.4.3. Sample Analysis Procedure

11.4.3.1. See Manufacturer's information for operating instructions.

11.4.3.2. A calibration curve is analyzed at the beginning of each run. The correlation coefficient must be > 0.995 to continue.

11.4.3.3. CCV's and CCB's are analyzed at the beginning, end, and between every 10 samples. CCV's must be prepared from a secondary source.

11.4.3.4. Sample distillates higher than the highest calibration standard (0.1 mg/L) must be diluted with 0.25 N NaOH and re-analyzed.

11.4.3.5. Any samples analyzed after a high sample must be re-analyzed if carryover is suspected.

11.5. Analytical Documentation

- 11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.
- 11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.
- 11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.
- 11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. *Total Cyanide, mg / L* =
$$\frac{\text{mg / L CN}^- \text{ from printout} \times 50}{\text{mL of sample distilled}} \times D$$

12.2. *Total Cyanide, mg / kg* =
$$\frac{\text{mg / L CN}^- \text{ from printout} \times 50}{\text{g of sample distilled.}} \times D$$

12.3. *Amenable Cyanide, mg / L* =
$$\text{Total CN}^- \text{ (mg / L)} - \text{Chlorinated CN}^- \text{ (mg / L)}$$

Where:

mg/L = *can also be mg/kg*

D = *Dilution Factor* =
$$\frac{\text{Final Volume of Dilution}}{\text{Volume of Sample Distillate Used}}$$

NOTE: Free cyanide has the same calculations as Total cyanide

12.4. LCS Recovery:

$$\frac{\text{Instrument Value}}{0.04(\text{true})} \times 100 = \% \text{ Recovery}$$

12.5. CCV Recovery:

$$\frac{\text{Instrument Value}}{0.025(\text{true})} \times 100 = \% \text{ Recovery}$$

NOTE: CCV recovery must be between 85 - 115% for data to be acceptable. If CCV recovery is not within these limits, reanalysis is required.

12.6. *MS/MSD Recovery for Waters and solids:*

$$\frac{A - B}{0.040(\text{true})} \times 100 = \% \text{ Recovery}$$

Where:

A = Instrument value MS/MSD

B = Sample instrument value

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. 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 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. All distillates can be rinsed down the drain with large amounts of water unless they contain high levels of Cyanide. If they are highly concentrated, they must be disposed of in the liquid waste container.
- 15.2. Solvent waste must be disposed of in clearly labeled waste cans.
- 15.3. Acid waste must be collected in clearly labeled acid waste containers.
- 15.4. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.
- 15.5. Refer to the Laboratory Sample and Waste Disposal plan.
- 15.6. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Quanterra. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

- 16.1. References
- 16.1.1. SW846. Test Methods for Evaluating Solid Waste, Third Edition. Update I; Total and Amenable Cyanide. Automated UV; Method 9012 A
- 16.1.2. EPA 600: Cyanide. Total and Cyanide. Amenable to Chlorination: Methods 335.1 and 335.2. March 1983
- 16.1.3. Standard Methods for the Examination of Water and Wastewater. Eighteenth Edition: Weak and Dissociable Cyanide; Method 4500-CN-E
- 16.1.4. USEPA CLP SOW ILM03.0, Section D - Cyanide Midi Distillation
- 16.1.5. Quanterra* Quality Assurance Management Plan, current revision

16.2. Associated SOPs

16.2.1. LM-WALN-1142 (NC-WC-0032), Cyanide Distillation Method

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) is 0.005 mg/L for waters (50 mL used) and 0.25 mg/kg for solids (1.0 g used).

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide for poor Base and Gain or baseline noise

17.2.1. Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes.

Place lines back in reagents and perform another base and gain when the baseline has been stabilized.

17.3. Method Deviation (9012A, 335.1, 335.2)

17.3.1. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

Appendix I - Cyanide Standardization

1. Pipet 10.0 mL of the 1000 ppm stock cyanide standard into a 250 mL Erlenmeyer flask and add 90 mL of reagent water.
2. Add 0.5 mL (10 drops) of Rhodanine indicator.
3. Titrate with 0.0192 N silver nitrate (using a micro buret) until the color changes from yellow to pink/orange.
4. Titrate a blank (100 mL reagent water) following steps 2 and 3.
5. Calculation:

$$\text{Cyanide, mg / L} = \frac{(A - B) (1000)}{\text{mL Cyanide Solution (10)}}$$

Where:

A = mL titrant for standard

B = mL titrant for blank

6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

Controlled Copy
Copy No: _____

UNCONTROLLED COPY

SOP No: CORP-OP-0001NC
Revision No: 3.1
Revision Date: 07/31/98
Implementation Date: 8/5/98
Page 1 of 73

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: CORP-OP-0001, Revision 3 (Dated 12/15/97))

Prepared by:	<u>Ruby Weber</u>	<u>7/31/98</u>
		Date
Reviewed by:	<u><i>[Signature]</i></u>	<u>7-4-98</u>
	Technology Specialist	Date
Approved by:	<u><i>[Signature]</i></u>	<u>8/4/98</u>
	Director, Quality Assurance	Date
Approved by:	<u><i>[Signature]</i></u>	<u>8-5-98</u>
	Director, Health and Safety	Date
Approved by:	<u><i>[Signature]</i></u>	<u>8-5-98</u>
	Management	Date
Approved by:	<u><i>[Signature]</i></u>	<u>7/2/98</u>
	Corporate Technology	Date

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Inc. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

The user agrees by its acceptance or use of this document to return it upon Quanterra's request and not to reproduce, copy, lend or otherwise dispose or disclose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties unless those parties also specifically agree to these conditions.

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 Preservation and Storage.....	10
9.	Quality Control	11
10.	Calibration and Standardization.....	13
11.	Procedure	13
11.1	Procedural Variations.....	13
11.2	Separatory Funnel Extraction from Aqueous Samples	14
11.3	Continuous Liquid/Liquid Extraction from Aqueous Samples	18
11.4	Sonication Extraction from Soil Samples.....	22
11.5	Soxhlet Extraction from soil samples	25
11.6	Waste dilution	27
11.6	Concentration.....	28
11.7	Cleanup Techniques.....	32
12.	Data Analysis and Calculations	47
13.	Method Performance.....	47
14.	Pollution Prevention	47
15.	Waste Management.....	47
16.	References.....	48
17.	Miscellaneous	48

List of Appendices:

Appendix A	Extraction Procedure for Chlorinated Acid Herbicides Based on Method 8150.....	64
Appendix B	Extraction Procedure for Chlorinated Acid Herbicides Based on Method 8151	75

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:
8080A, 8100, 8140, 8141A, 8150B, 8151A, 8270B, 8310
608, 610, and 625
- 1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

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.
- 2.4. Soxhlet Extraction
A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent.
- 2.5. Cleanup and Concentration
Procedures are presented for removing interferences 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

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 that lead to discrete artifacts. 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 appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become 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]. When good manual dexterity is needed, for example, when handling small quantities/containers, disposable gloves (such as latex or N-DEX®) shall be used. While these gloves protect against splashes, they give little or no protection against contact with large quantities of solvent, and no protection against spills or immersion.
- 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:

Chemicals that have been classified as **carcinogens** or **potential carcinogens** under OSHA include: Methylene chloride, PCBs, certain pesticides.

The following materials are known to be **corrosive**: Concentrated sulfuric acid, sodium hydroxide, 1N nitric acid.

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.

Chemicals known to be **flammable** are: Diethyl ether, acetone, hexane, 2-propanol.

Mercury is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and clean-up techniques before handling this material.

- 5.4. 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.
- 5.5. 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.
- 5.6. 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.

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.

6.2. Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep fun	Cl.LH	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)	√		√		

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
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					√
<i>Jacketed Concentrator Tube</i>					√
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	√	√	√	√	√
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).	√				

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid	A/B
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.	Cl LF	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 ₂ SO ₄), Concentrated: Reagent Grade	√	√			
Sulfuric acid (1:1): Carefully add 500 mL of H ₂ SO ₄ to 500 mL of reagent water. Mix well.	√	√			
Organic free reagent water.	√	√			
Sodium sulfate (Na ₂ SO ₄), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√	√	√	
Extraction/Exchange Solvents: Methylene chloride, hexane.	√	√	√	√	√

REAGENTS	Sep fun.	CLLE	Soni	Sox	Conc
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 (catalog 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 ₂ SO ₄). Concentrated: Reagent Grade					√
Sulfuric acid (1:1): Carefully add 500 mL of H ₂ SO 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 neat. Semivolatile stock standards are stored at $\leq 6^{\circ}\text{C}$. All stock standards must be protected from light. Stock standard solutions must be replaced after six months (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.

7.3.2. 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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ 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.

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 as a nonconformance.

9.4. Sample count

Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field QC samples and multiple dilutions of 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 *approximately* 90 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 but spiked with the LCS standard and the surrogates (See sections 9.5.1 - 9.5.3).
- 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

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. There are slight differences in the initial steps for SW-846 and 600 series methods, as defined below.

11.2.1. Sample preparation for SW-846 methods

- 11.2.1.1. 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.
- 11.2.1.2. Add 60 mL of methylene chloride directly to the separatory funnel. Transfer the sample to the separatory funnel. The sample volume is determined by marking the meniscus level on the sample container. Mark the meniscus and transfer the sample to the separatory funnel. Fill the sample container with water up to the marked level. Transfer the measured amount of water to a graduated cylinder and record the volume on the benchsheet to the nearest 5 mL. Less than one liter of sample may be used. If necessary, dilute the sample to about 1 liter with reagent water.
- 11.2.1.3. 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.
- 11.2.1.4. Use 100 mL of leachate for TCLP pesticides, and 200 mL of leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate should be made up to 1 L in volume with reagent water.
- 11.2.1.5. 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. Add 60 mL of methylene chloride to the separatory funnel.

11.2.1.6. Proceed to section 11.2.3 (SW-846 and 600 series).

11.2.2. 600 Series methods

- 11.2.2.1. Mix the sample well by shaking the container.
- 11.2.2.2. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet.
- 11.2.2.3. Add 60 mL of methylene chloride directly to the separatory funnel. Transfer the sample to the separatory funnel. Empty the container

completely. Add *approximately 10-20 mL* of methylene chloride to the sample container, seal and shake to rinse the inner surface and remove residual analytes adhering to the glass walls. Transfer this solvent to the separatory funnel.

- **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. The sample volume may also be determined by marking the meniscus level on the sample container. Transfer the sample to the extractor (see 11.1.3). Fill the sample container with water up to the marked level. Transfer the measured amount of water to a graduated cylinder and record the volume on the benchsheet to the nearest 5 mL.
- Less than one liter of sample may be used. Dilute the sample to about 1 liter with reagent water.

11.2.2.4. 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.

11.2.2.5. Proceed to section 11.2.3 (SW-846 and 600 series)

11.2.3. Add 1 mL of the surrogate spiking solution (Table 3) to each sample, method blank, Laboratory Control Sample (LCS), and Matrix Spike/Matrix Spike Duplicate (MS/MSD). Add 1 mL of the matrix spiking solution (Table 4) to each LCS and MS/MSD. Note that different matrix spiking solutions are used for 600 series, 8000 series and TCLP batches. Swirl liquid in separatory funnel gently to homogenize sample and surrogate/spiking compounds. Note that the surrogate and matrix spiking solutions should be brought to room temperature before spiking the samples.

11.2.4. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H₂SO₄ 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.

11.2.5. Seal and shake the separatory funnel vigorously for 1-2 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.

11.2.6. 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 11.2.). 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.

11.2.7. 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. *Add a small amount of methylene chloride to wet 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.

11.2.8. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.9. 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 H₂SO₄. 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 11.1.7 to 11.1.9. 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.

11.2.10. 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.

11.2.11. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

- 11.2.12. 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. There are slight differences in the initial steps for SW-846 and 600 series methods, as defined below.

11.3.1. Sample preparation for SW-846 methods.

11.3.1.1. Add 3 to 5 boiling chips to the round bottom distilling flask. Assemble to the *liquid/liquid body*. Add 200-300 mL of methylene chloride to the extractor body.

Note: Other liquid/liquid designs may require the addition of different amounts of methylene chloride.

11.3.1.2. 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.

11.3.1.3. The sample volume is determined by marking the meniscus level on the sample container. Transfer the sample to the extractor (see 11.1.3). Fill the sample container with water up to the marked level. Transfer the measured amount of water to a graduated cylinder and record the volume on the benchsheet to the nearest 5 mL.

11.3.1.4. 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.

11.3.1.5. Use 100 mL of leachate for TCLP *herbicides*, and 250 mL of leachate for TCLP *pesticides and semivolatiles*, measured in a graduated cylinder. The leachate may be made up to 1 L in volume with reagent water.

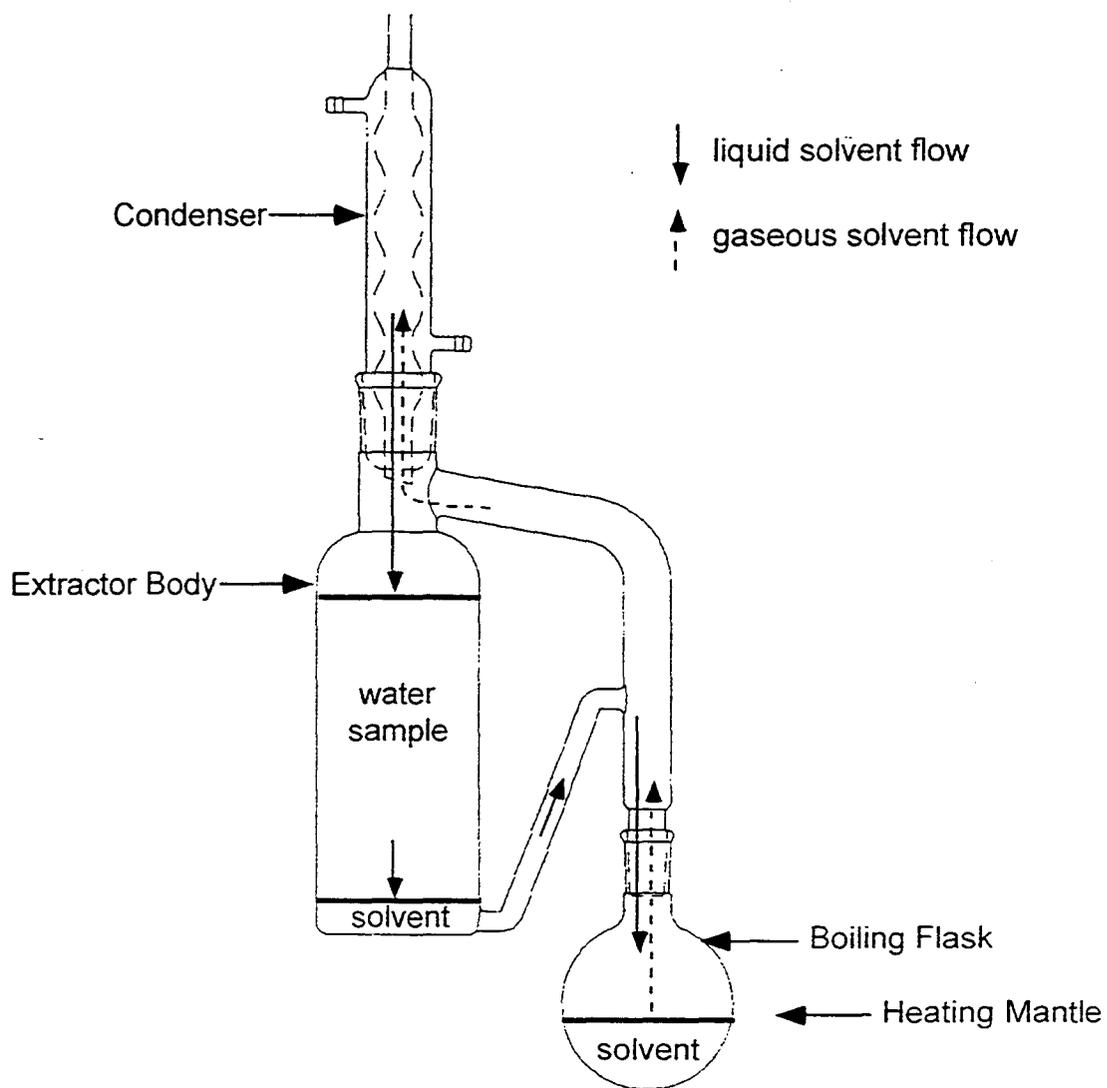
11.3.1.6. For a TCLP method blank, measure 100 mL (*for herbicides*) or 250 mL (*for pesticides and semivolatiles*) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water.

11.3.1.7. 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.

11.3.1.8. Proceed to section 11.3.3 (SW-846 and 600 series methods).

11.3.2. Sample preparation for 600 series methods.

- 11.3.2.1. Add 3 to 5 boiling chips to the round bottom distilling flask. Assemble *to the liquid/liquid body*. Add 200 mL of methylene chloride to the extractor body.
- 11.3.2.2. Mix the sample well by shaking the container.
- 11.3.2.3. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet.
- 11.3.2.4. The sample volume *is* determined by marking the meniscus level on the sample container. Transfer the sample to the extractor (see 11.1.3). Fill the sample container with water up to the marked level. Transfer the measured amount of water to a graduated cylinder and record the volume on the benchsheet to the nearest 5 mL. Add *approximately* 20 mL of methylene chloride to the sample container, seal and shake to rinse the inner surface and remove residual analytes adhering to the glass walls. Transfer this solvent to the continuous extractor body. If the entire sample is not used the sample volume is measured using a graduated cylinder.
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.
- 11.3.2.5. 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.
- 11.3.2.6. 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.
- 11.3.2.7. Proceed to section 11.3.4 (SW-846 and 600 series methods).



11.3.3. Add 1 mL of the surrogate spiking solution (Table 3) to each sample, method blank, Laboratory Control Sample (LCS), and Matrix Spike/Matrix Spike Duplicate (MS/MSD). Add 1 mL of the matrix spiking solution (Table 4) to each LCS and MS/MSD.

- **Different matrix spiking solutions are used for 600 series, 8000 series and TCLP batches.**
- Surrogate and matrix spiking solutions should be brought to room temperature before spiking the samples.

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. Attach cold condenser (about 10°C) to the extractor body. 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 H₂SO₄. 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 nor 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 appropriate waste container.
- 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

11.4.1. A flow chart for this procedure is included in Section 17.

11.4.2. Determination of percent moisture (Optional - if a different group performs this test, refer to the facility SOP.)

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 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.4.3. Determination of pH (Optional - if a different group performs this test, refer to the facility SOP.)

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.

11.4.4. Sonication Procedure

11.4.4.1. 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.

11.4.4.2. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.4.4.3. Weigh 30 g of sample ± 0.2 g into a 250 or 400 mL beaker. Record the weight to the nearest 0.1 g in the appropriate column on the benchsheet. Use *approximately* 90 g of sodium sulfate for the method blank and LCS.

11.4.4.4. Mix weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 60 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.4.5. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP.

11.4.4.6. Add 1 mL of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add 1 mL of the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Table 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.

11.4.4.7. Immediately add a minimum of 100 mL of 1:1 methylene chloride:acetone to the beaker.

Note: Use methylene chloride only if the extract is for Diesel Range Organics or similar tests that require determination of the total area of the chromatogram by GC-FID. If acetone is used the aldol condensation product will interfere with these tests.

Note: Steps 11.4.4.2 - 11.4.4.6 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.4.8. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.4.9. 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 ¾ inch high gain (Q) horn or 10 for the ¾ inch standard horn. with mode switch on pulse. and percent-duty cycle knob set at 50%.

Note: Do *not* use *Microtip* probe.

11.4.4.10. 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 anhydrous sodium sulfate to the funnel cup.

11.4.4.11. Place the prepared funnel on a collection apparatus (beaker or K-D Apparatus).

11.4.4.12. Decant and filter extracts through the prepared funnel into a clean beaker or K-D Apparatus.

11.4.4.13. 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 sodium sulfate.

11.4.4.14. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4.5. Sonicator Tuning.

11.4.5.1. 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

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

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 ± 0.2 g into a beaker, recording the weight to the nearest 0.1 g on the benchsheet. Use *approximately* 30 g of sodium sulfate for the method blank and LCS. Add 30 g of anhydrous sodium sulfate 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.

Note: If the sample is visibly dry, weigh directly into a thimble and mix with approximately 30 g of sodium sulfate.

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 1 mL of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add 1 mL of 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 200 mL of 1:1 methylene chloride:acetone into a 250 mL *round* bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Note: Use methylene chloride only if the extract is for Diesel Range Organics or similar tests that require determination of the total area of the chromatogram by GC-FID. If acetone is used the aldol condensation product will interfere with these tests.

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 $1.0 \text{ g} \pm 0.1 \text{ g}$ of sample to the vial. Record the weight to the nearest 0.1 g.
- 11.6.4. Add 1 mL of surrogate solution (Table 3).
- 11.6.5. Dilute to 10 mL (make up to the meniscus mark) with the appropriate solvent. (Methylene Chloride for GC/MS analysis, hexane for pesticides analysis.)
- 11.6.6. Add $2 \text{ g} \pm 0.1 \text{ g}$ sodium sulfate to the sample. Cap and *vortex* for 2 minutes.
- 11.6.7. *Optional:* Add 4-5 g sodium sulfate to a small funnel. The funnel can be plugged with glass wool 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.
Note: Concentrate extracts for GC/MS analysis to 2 mL.
- 11.6.9. Label the sample. which is now ready for cleanup or analysis.

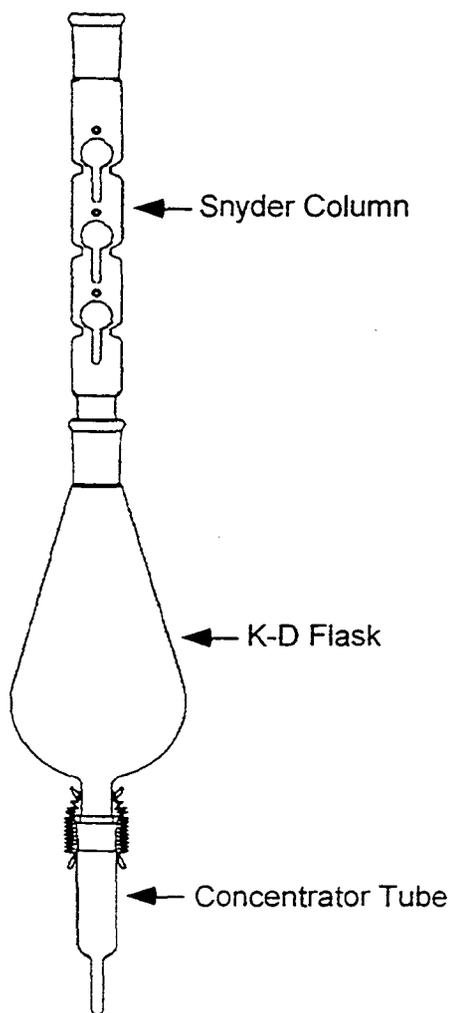
11.7. Concentration

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) Methods:

11.7.1.1. *Water Bath-Based Concentration Method:*

- 11.7.1.1.1. 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.
- 11.7.1.1.2. 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).



- 11.7.1.1.3. 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.
- 11.7.1.1.4. 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.
- ~~11.7.1.1.5.~~ 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.

11.7.1.1.6. *Note:* It is very important not to concentrate to dryness as analytes will be lost.

11.7.1.1.7. 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.

11.7.1.1.8. 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

11.7.1.2. Alternative QES Concentration Method:

This concentration method uses a hot water jacketed concentrator tube (jCT) instead of the hot water bath and concentrator tube used in Section 11.7.1.1. The construction of the jacketed concentrator tube reduces the tendency of the extract to evaporate to dryness. Thus, low boiling analytes are retained in the extract better with less analyst monitoring of the concentration process.

11.7.1.2.1. *Assemble the jacketed concentrator tube, KD body and hot water hoses. Add 1 mL of exchange solvent (if needed) and one large, clean boiling chip.*

NOTE: The boiling chips used in the jacketed concentrator tube must be large enough to prevent them falling down into the tip of the jCT. If the boiling chip is not in the proper position, the extract may superheat and bump vigorously if the extract solvent warms up slowly.

11.7.1.2.2. *Pour the extract to be concentrated into the KD flask, and attach a three-ball Snyder column.*

11.7.1.2.3. *Turn on the hot (90 °C) water flow to the jacketed concentrator tube.*

11.7.1.2.4. *Concentrate to 1 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent (5-10 mL hexane, 1*

mL acetonitrile, 4 mL acetone/hexane) to the top of the Snyder column. Continue to concentrate until the Snyder column balls stop chattering.

11.7.1.2.5. *Cool the jacketed concentrator tube until it is cold to the touch.*

11.7.1.2.6. *Quantitatively transfer the extract and dilute to final volume, or continue concentration with nitrogen evaporation (section 11.7.2).*

11.7.2. Nitrogen Evaporation to Final Concentration

11.7.2.1. Place the *concentrator* tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

11.7.2.2. *Evaporate the extract to below the final volume line, using the concentrator tube calibration line as a guide; and remove the CT from the water bath. Rinse the sides of the concentrator tube with the extract, using a pipet, and transfer the extract to the appropriate storage vial. Add a small amount of the appropriate solvent to the concentrator tube and rinse again. Bring the extract to final volume with the solvent rinse, using a measured vial for side-by-side comparison.*

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.7.2.3. 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.

11.7.2.4. **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.
- 11.7.3.7. *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.
- 11.7.3.8. 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.
- 11.7.3.9. During the concentration rinse the Turbovap tube walls with a few mL of solvent 1 or 2 times.
- 11.7.3.10. Concentrate the extract to slightly less than the required final volume.
- 11.7.3.11. 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.
- 11.7.3.12. 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 8080 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 8080. In this case the GPC should be performed first.

Sulfur cleanup (Section 11.8.3) is generally applied to samples for analysis by method 8080, 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 Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by the sulfuric acid.

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

FROM WATERS AND SOILS, BASED ON SW-846 3500 SERIES,
3600 SERIES, 8150, 8151 AND 600 SERIES METHODS.

Revision No: 2.4
Revision Date: 10/16/98
Page 36 of 95

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 11.8.1.6):

- 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 $\pm 5\%$ 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 85%. 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.

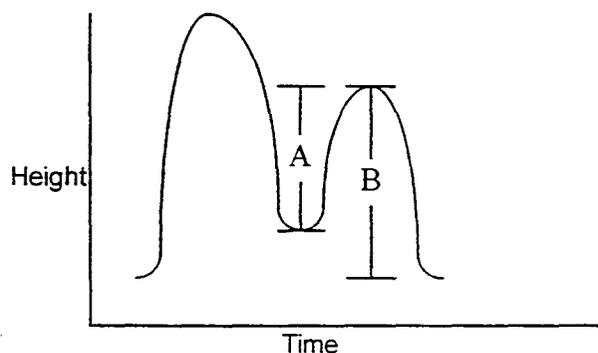
11.8.1.6.2. Start the pump and let the flow stabilize for 2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to

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

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/arocloris 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 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.

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 pipet 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. Properly dispose of the mercury waste.

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 7.1.15.4 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. *Vortex until sample and acid are thoroughly mixed* 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. Properly dispose of the acid waste.

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. Adjust the aqueous phase to a pH of 1-2 with sulfuric acid (1:1). Allow to cool and 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 appropriate waste container.

11.8.6. Silica Gel Cleanup

This procedure is appropriate for cleanup of extracts prior to determination of polynuclear aromatic hydrocarbons by GC/FID.

- 11.8.6.1. Exchange the extract solvent to cyclohexane and reduce the volume to 2 mL prior to cleanup.
- 11.8.6.2. 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.
- 11.8.6.3. Preelute 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 elute.
- 11.8.6.4. 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.

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.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated, and disposed according to the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

15.1. Solvent waste must be disposed of in the clearly labeled waste cans.

15.2. Acid waste must be collected in the clearly labeled acid waste containers.

- 15.3. Solid materials (soil, gloves, soiled paper products) are placed in the solid debris container. Do not put liquids in the solid waste container.
- 15.4. Methylene chloride saturated water and remaining sample after the extraction is dumped into Methylene chloride waste cans.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II (September 1994). Sections 3500A, 3510B, 3520B, 3540B, 3550A, 3600B, 3610A, 3620A, 3640A, 3650A, and 3660A.

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. In the CLLE procedure the same solvent is used for the acid and base neutral extractions.
- 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. Sonicator tuning directions have been changed
- 17.2.2. Use of phase separation filter paper is not recommended, since organic solvents may leach siloxanes from the filter paper.
- 17.2.3. The quantity of sulfuric acid used in sulfuric acid cleanup has been reduced.
- 17.2.4. Allowance for different final solvents for organophosphorous pesticides has been added.
- 17.2.5. Some typographical errors have been corrected.

17.3. Facility Specific SOPs

17.3.1. 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.

17.3.2. *Refer to the SOP change form on file in the North Canton Quality Assurance Department.*

17.3.3. *Deviation from Revision 2: Section 11.7.2.2*

The extract is not concentrated to its final volume in the concentrator tube. It is transferred to the appropriate storage vial, brought up to volume with solvent rinse from the concentrator tube, and visually checked against the measured volume in another vial. A comparison study of measured samples versus samples bottled by visual comparison to a measured vial indicates no significant difference between the analyte recoveries of the alternative procedures. Data related to this study is available upon request in the Quality Assurance department.

17.3.4. *Modification from previous revision 2.2, dated 04/18/94*

Addition of Methylene chloride first to the extraction apparatus is due to a limitation of almost all CLLE glassware. If the sample is added first, then sample water will fill the transfer tube which connects the bottom of the continuous extractor body to the boiling flask. If Methylene chloride is now added, all the sample water in the transfer tube will be pushed into the boiling flask. This can cause nonextractable sample components to end up in the extract. It can also interfere with the recovery of polar analytes (e.g. phenols, amines) since these analytes may "extract" out of the solvent in the boiling flask and back into this "misplaced" sample water.

17.4. Tables

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270	11-12	1-2
625	11-12	1-2
Pest/PCB: 8080 & 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

NOTE: *For traditional TCL 8270 list, an acid only extraction is performed.*

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	N/A	1.0
Pesticides	Hexane	10.0
PAH by HPLC	Acetonitrile	Refer to facility SOP for HPLC
PAH by GC	N/A	1.0
TPH	N/A	1.0
OPP	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	CH ₂ Cl ₂	10 mL ¹	0.5 mL CH ₂ Cl ₂
Pesticides	CH ₂ Cl ₂	10 mL ¹	5 mL, hexane
OPP	CH ₂ Cl 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	Hexane	10 mL (2 mL aliquot used)	2 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	Methylene Chloride	10 mL	Hexane	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.

Table 3		
Surrogate Spiking Solutions		
Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.5
Pest	0.2 ppm DCB/TCX	1.0
Pest/PCB	0.2 ppm DCB/TCX	1.0
PCB	0.2 ppm DCB/TCX	1.0

Table 4		
Matrix Spike and LCS Solutions		
Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.5
BNA TCLP	BNA TCLP Spike	1.0
BNA NPDES	BNA NPDES Spike	1.0
Pest	0.2/0.5 ppm Pest	1.0
Pest TCLP	Pest TCLP Spike	1.0
Pest NPDES	Pest NPDES Spike	1.0
Pest/PCB	0.5 ppm pest spike	1.0
PCB	10 ppm PCB Spike	1.0

Table 5			
Surrogate Spike Components			
Type	Compounds	Solvent	Conc. ($\mu\text{g/mL}$)
BNA	2-Fluorobiphenyl	Methanol	50
	Nitrobenzene-d5		50
	p-Terphenyl-d14		50
	2-Fluorophenol		75
	Phenol-d6		75
	2,4,6-Tribromophenol		75
	1,2-Dichlorobenzene-d4		50
	2-Chlorophenol-d4		75
Pest/PCB	Decachlorobiphenyl	Acetone	0.2
	Tetrachloro-m-xylene		0.2

Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. ($\mu\text{g/mL}$)
TCL BNA	Acenaphthene	Methanol	50
	4-Chloro-3-Methylphenol		75
	2-Chlorophenol		75
	1,4-Dichlorobenzene		50
	2,4-Dinitrotoluene		50
	4-Nitrophenol		75
	N-Nitroso-Di-n-Propylamine		50
	Pentachlorophenol		75

Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. ($\mu\text{g/mL}$)
	Phenol		75
	Pyrene		50
	1,2,4-Trichlorobenzene		50
BNA	1,4-Dichlorobenzene	Methanol	50
TCLP	2,4-Dinitrotoluene		50
	Hexachlorobenzene		50
	Hexachlorobutadiene		50
	Hexachloroethane		50
	2-Methylphenol		50
	3-Methylphenol		50
	4-Methylphenol		50
	Nitrobenzene		50
	Pentachlorophenol		50
	Pyridine		50
	2,4,5-Trichlorophenol		50
	2,4,6-Trichlorophenol		50
BNA NPDES		Methanol	
	Acenaphthene		50
	Acenaphthylene		50
	Anthracene		50
	Benzo(a)anthracene		50
	Benzo(b)fluoranthene		50
	Benzo(k)fluoranthene		50
	Benzo(a)pyrene		50

Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. ($\mu\text{g/mL}$)
	Benzo(ghi)perylene		50
	Benzyl butyl phthalate		50
	Bis(2-chloroethyl)ether		50
	Bis(2-chloroethoxy)methane		50
	Bis(2-ethylhexyl)phthalate		50
	Bis(2-chloroisopropyl)ether		50
	4-Bromophenyl phenyl ether		50
	2-Chloronaphthalene		50
	4-Chlorophenyl phenyl ether		50
	Chrysene		50
	Dibenzo(a,h)anthracene		50
	Di-n-butylphthalate		50
	1,3-Dichlorobenzene		50
	1,2-Dichlorobenzene		50
	1,4-Dichlorobenzene		50
	3,3'-Dichlorobenzidine		50
	Diethyl phthalate		50
	Dimethyl phthalate		50
	2,4-Dinitrotoluene		50
	2,6-Dinitrotoluene		50
	Di-n-octylphthalate		50
	Fluoranthene		50
	Fluorene		50
	Hexachlorobenzene		50

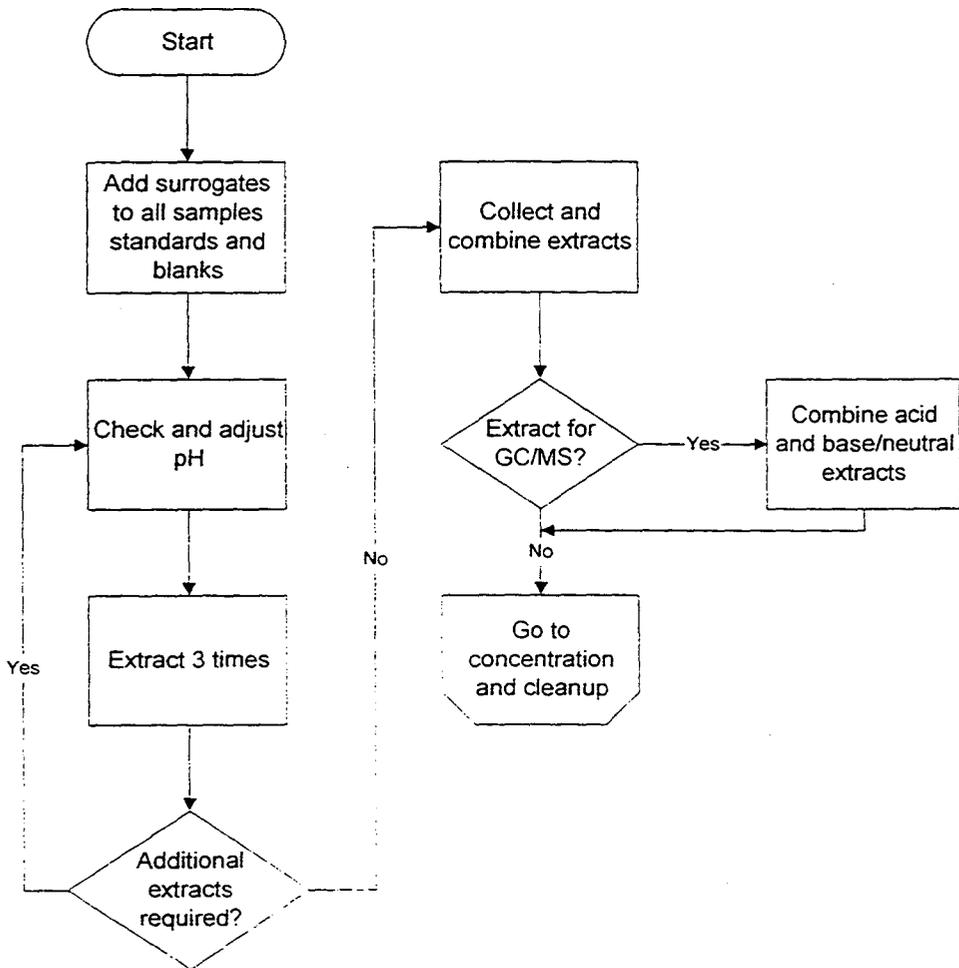
Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. (µg/mL)
	Hexachlorobutadiene		50
	Hexachloroethane		50
	Indeno(1,2,3-cd)pyrene		50
	Isophorone		50
	Naphthalene		50
	Nitrobenzene		50
	N-Nitrosodi-n-propylamine		50
	Phenanthrene		50
	Pyrene		50
	1,2,4-Trichlorobenzene		50
	4-Chloro-3-methylphenol		50
	2-Chlorophenol		50
	2,4-Dichlorophenol		50
	2,4-Dimethylphenol		50
	2,4-Dinitrophenol		50
	2-Methyl-4,6-dinitrophenol		50
	2-Nitrophenol		50
	4-Nitrophenol		50
	Pentachlorophenol		50
	Phenol		50
	2,4,6-Trichlorophenol		50
Pest	Aldrin	Acetone	0.2
	gamma-BHC (Lindane)		0.2
	4,4'-DDT		0.5

Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. ($\mu\text{g/mL}$)
	Dieldrin		0.5
	Endrin		0.5
	Heptachlor		0.2
Pest TCLP	Heptachlor	Acetone	0.5
	Heptachlor epoxide		0.5
	Lindane		0.5
	Endrin		0.5
	Methoxychlor		1.0
Pest NPDES	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
	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

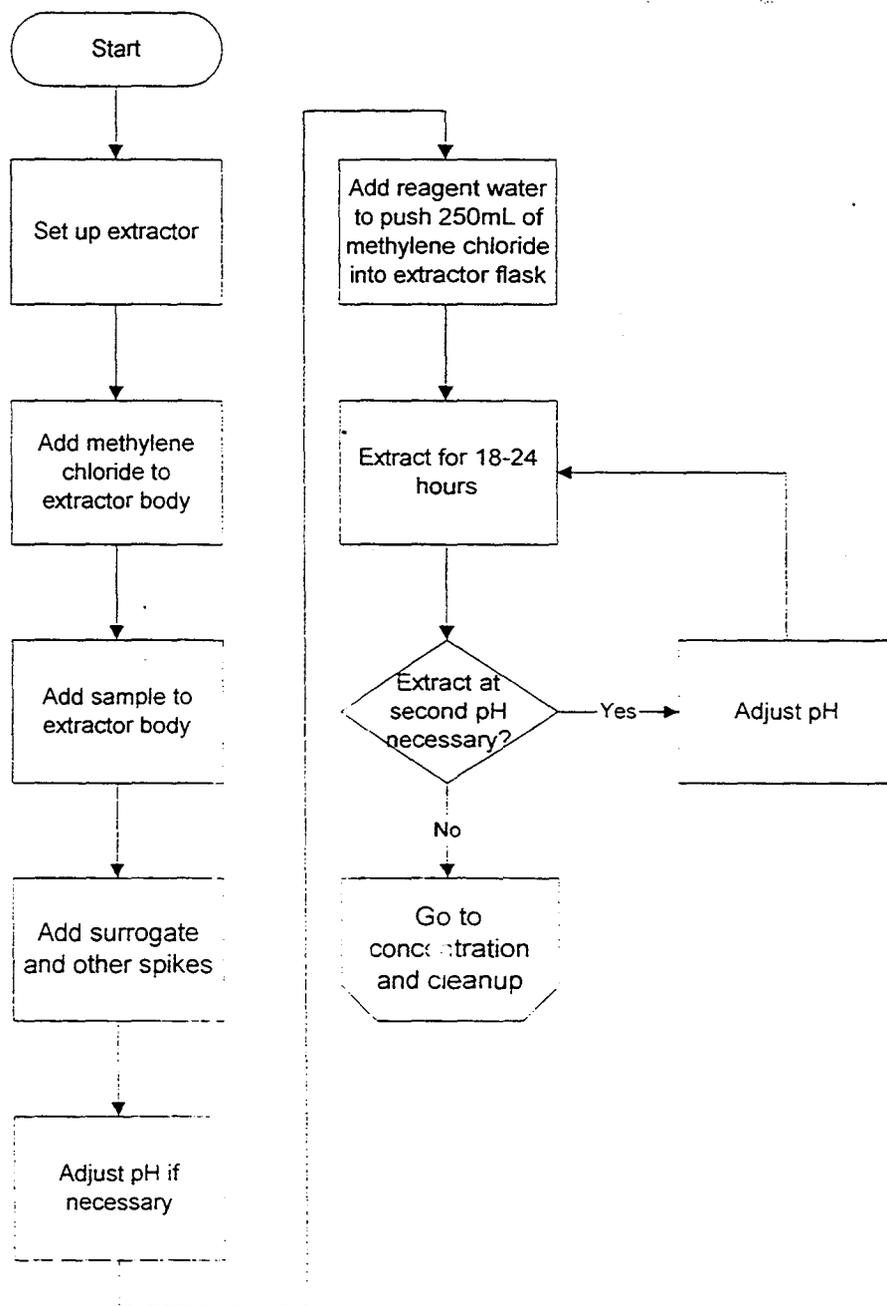
Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. ($\mu\text{g}/\text{mL}$)
PCB only	PCB 1016/1260	Acetone	10

17.5 Flow diagrams

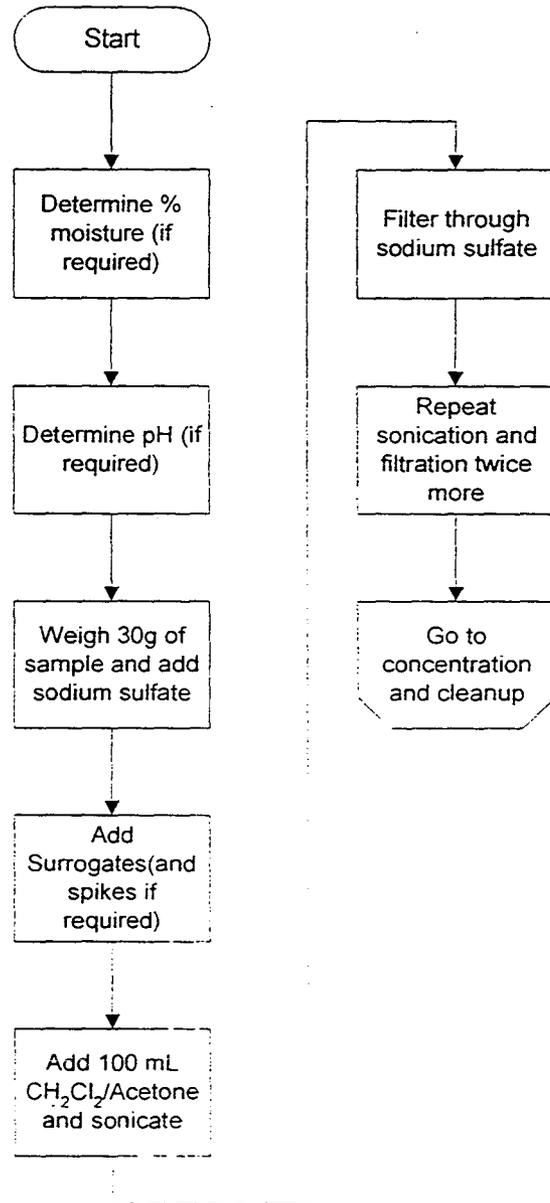
17.4.1. Separatory funnel extraction



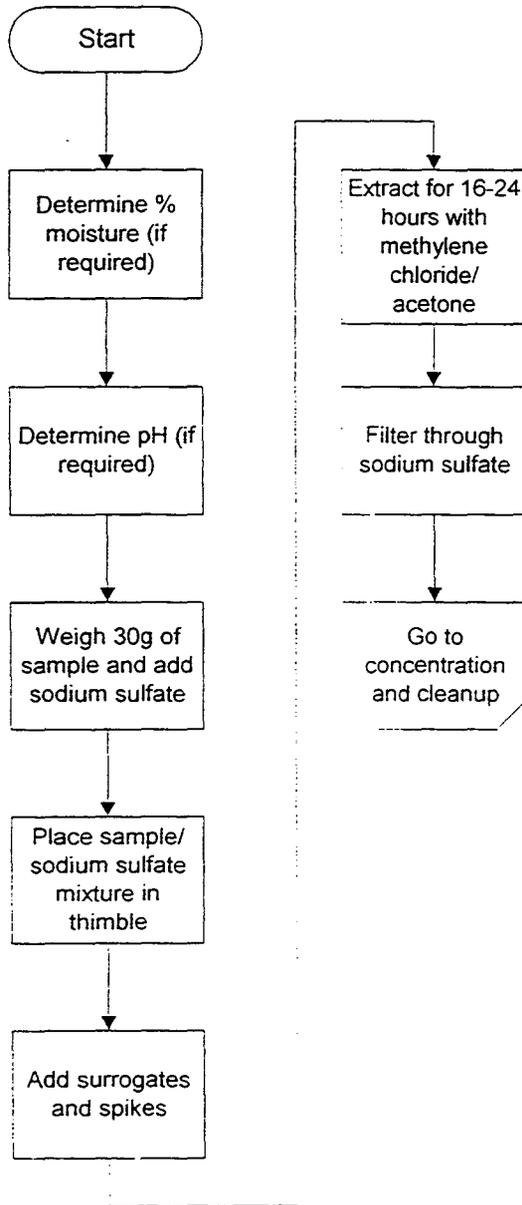
17.4.2. Continuous liquid/liquid extraction



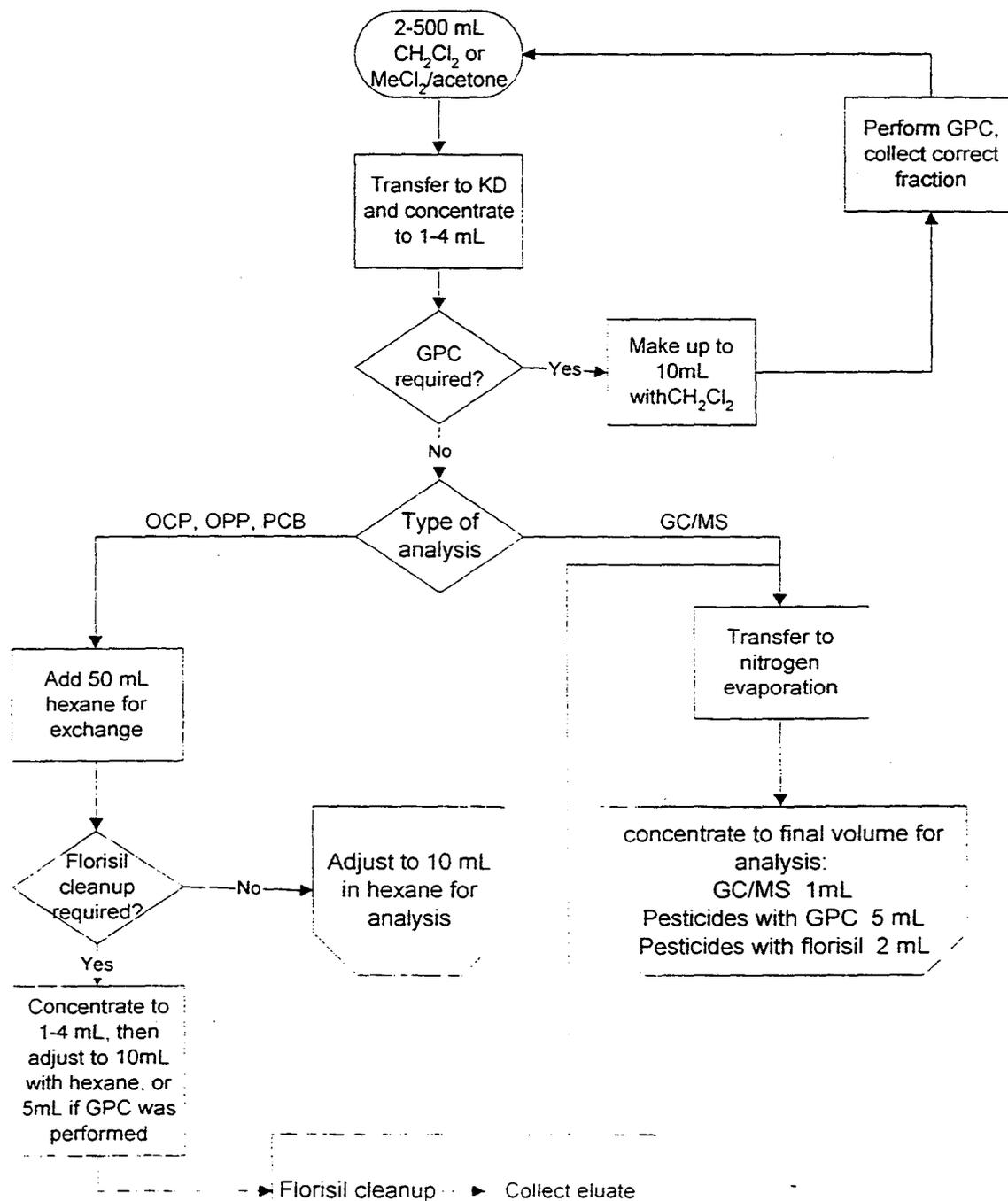
17.4.3. Sonication Extraction



17.4.4. Soxhlet extraction



17.4.5. 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 8150B.

2. SUMMARY OF METHOD

This method is based on SW846 method 8150B. Aqueous samples are extracted into diethyl ether by a separatory funnel extraction. Solids are extracted into diethyl ether by shaking. Phenoxy acid herbicides in the extract are hydrolyzed with potassium hydroxide and the aqueous solution is washed with diethyl ether to remove interferences. The aqueous solution is then acidified and the herbicides are extracted with diethyl ether. The ether solution is dried and the herbicides are esterified using diazomethane. 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
- 5.4. Diethyl ether must be free of peroxides as demonstrated by EM Quant test strips.
- 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.

7.2.2. Diazald, 99% purity

7.2.3. 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 slurring 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.

7.2.4. 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).

7.2.5. Diethyl ether, reagent grade.

7.2.6. Methanol, reagent grade.

7.2.7. 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 soil, sediment and other solid samples

11.1.1. Weigh 50.0 g of moist solid sample into an 8 oz. glass jar. Use 50 g of sodium sulfate for the Method Blank and LCS. Acidify the sample with 5 mL of concentrated HCl.

11.1.2. 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)

11.1.3. 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 is not determined.)

11.1.4. Add 60 g of acidified sodium sulfate and mix well. The sample should be free flowing. If not, add more sodium sulfate.

11.1.5. Spike with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

11.1.6. Add 100 mL 20% acetone in ethyl ether.

Note: If dinoseb is a target, acetone should not be used. Instead, use 100 mL of ethyl ether.

11.1.7. Mix contents on orbital shaker for 20 minutes. Decant extract through glass wool plugged funnel.

-
- 11.1.8. Extract sample twice more, using 100 mL 20% acetone in ether (or 100% ether if dinoseb is a target). After each addition shake for 10 minutes and decant the extract.
 - 11.1.9. Combine the extracts in a 1 or 2 liter separatory funnel containing 250 mL of acidified 5% sodium sulfate solution.
 - 11.1.10. Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at the desired pH. Gently mix the contents of the separatory funnel for one minute to extract interferences into the aqueous layer. Allow the layers to separate. Collect the aqueous phase in a clean beaker and the extract phase in a 500 mL ground glass Erlenmeyer flask. Place the aqueous phase back in the separatory funnel and re-extract using 25 mL of diethyl ether. Allow the layers to separate and discard the aqueous layer. Combine the ether extracts in the 500 mL Erlenmeyer flask containing 5 mL of 37% potassium hydroxide. Proceed to step 11.3. Hydrolysis.

11.2. Preparation of Aqueous Samples

- 11.2.1. Weigh the sample bottle and pour approximately 1 liter (100 mL for TCLP leachates) into a 2 liter separatory funnel. The sample should be decanted off any sediment. 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.
- 11.2.2. Adjust pH to 2 with 1:1 sulfuric acid. Spike with 1 mL of surrogate solution. Spike MS/MSD and LCS samples with 1 mL of matrix spiking solution. (Refer to Tables A1 and A2). Add 150 mL diethyl ether and shake funnel for 2 minutes with frequent venting to release excess pressure. **Caution:** Diethyl ether will generate pressure rapidly. Vent the funnel immediately after it is first sealed and inverted, and vent frequently thereafter.
- 11.2.3. Allow layers to separate for at least 10 minutes and drain the aqueous phase into a clean beaker. If an emulsion forms break by centrifuge or mechanically. Collect the solvent extract (upper layer) in a 500 mL Erlenmeyer containing 2 mL of 37% potassium hydroxide. Use 5 mL of potassium hydroxide for TCLP samples.
- 11.2.4. Repeat the extraction two more times using 50 mL of diethyl ether each time. Combine the extracts in the Erlenmeyer flask. Rinse the beaker with each additional aliquot of extraction solvent. Proceed to section 11.3. Hydrolysis.

11.3. Hydrolysis

- 11.3.1. Add one or two clean boiling chips, the sample extract, and 15 mL of water for water samples (30 mL for soil samples) to the 500 mL flask. Attach a three-ball Snyder column.
- 11.3.2. Place the apparatus on a hot water bath (approx 60°C) so that the bottom of the flask is bathed with hot water vapor. Although the diethyl ether will evaporate in about 15 minutes, continue heating for a total of 60 minutes), beginning from the time the flask is placed in the water bath. Remove the apparatus and let stand at room temperature for at least 10 minutes. Check the pH of the solution. If not at or above 11, add additional KOH to bring the pH above 11 and hydrolyze for an additional 60 minutes.

11.4. Solvent Clean-up

- 11.4.1. Transfer the solution to an acid-rinsed 125 or 250 mL separatory funnel using 5-10 mL of reagent water to rinse out the flask. Wash the basic solution by shaking for 1 minute with a 20 mL portion of diethyl ether. Drain the aqueous (bottom) layer back into the flask and discard the ether layer. Pour the aqueous layer back into the separatory funnel and repeat the wash with a second 20 mL of ether. Pour the aqueous phase back into the separatory funnel. The herbicides remain in the aqueous phase. Additional washes may be used if the sample appears dirty.

11.5. Solvent Extraction

- 11.5.1. Acidify the contents of the separatory funnel to pH 2 by adding 1:1 sulfuric acid (2 mL for aqueous, 5 mL for soils). Test with pH indicator paper. Add 40 mL ether and shake vigorously for 1 minute. Drain and collect the aqueous phase and pour the ether phase into a flask or bottle containing 5-7 g of acidified sodium sulfate.
- 11.5.2. Pour the aqueous phase back into the separatory funnel and repeat the extraction twice more with 20 mL aliquots of ether, combining all solvent in the flask or bottle. Allow the extract to remain in contact with the sodium sulfate for at least 2 hours.

NOTE: The drying step is very critical to ensure complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all the sodium sulfate solidifies in a cake, add a few additional grams of sodium sulfate and again test by swirling. The 2 hour

drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

11.6. Concentration

- 11.6.1. Transfer the ether extract by decanting, or through a funnel plugged with acid washed glass wool, 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 Erlenmeyer with 20-30 mL ether to complete transfer.
- 11.6.2. 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 2-5 mL, remove from the water bath and allow to completely cool.
- 11.6.3. Carefully disassemble the concentrator tube and rinse the lower glass joint with a small amount of diethyl ether.
- 11.6.4. Add 0.1 mL of methanol.
- 11.6.5. The extract is now ready for esterification by either the diazomethane solution method (11.7) or the bubbler method (11.8)

11.7. Esterification (diazomethane solution method)

11.7.1. Preparation of Diazomethane solution

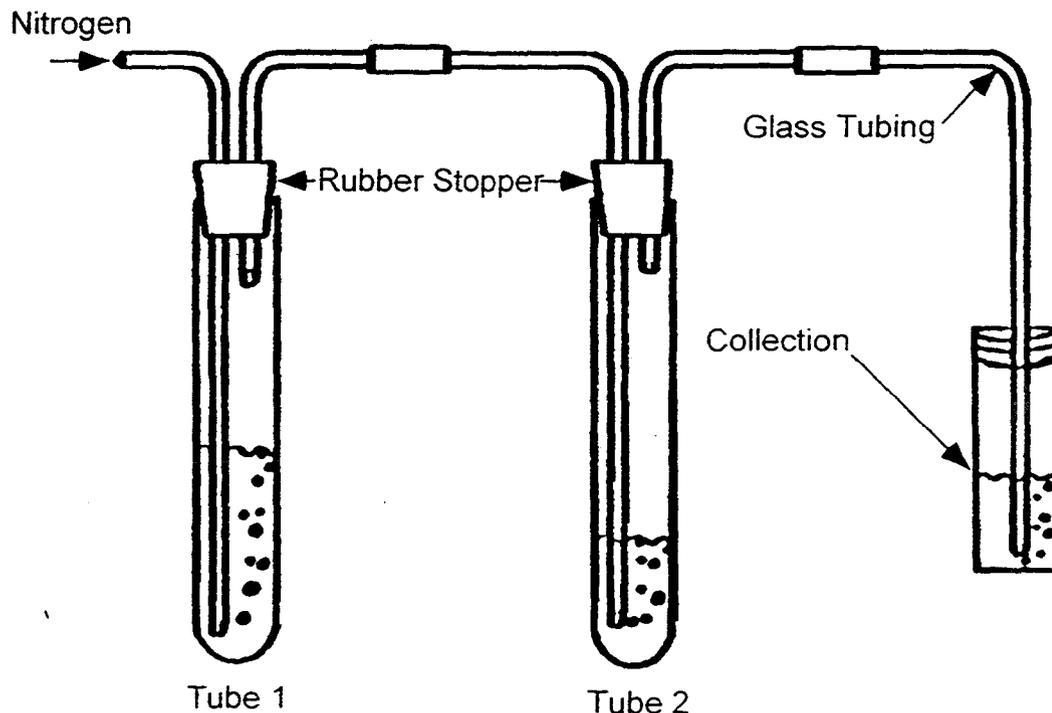
- **CAUTION: Diazomethane is potentially explosive.**
- **A temporary shield or the sash of the hood must protect the face and body of the analyst.**
- **Never heat solutions of diazomethane above 90°C, due to the explosive potential.**
- **Do not use glass stirring rods or any glassware with ground 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.**

- 11.7.1.1. 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.

- 11.7.1.2. Attach a 100 mL receiving flask to the condenser and cool the receiver in an ice bath.
- 11.7.1.3. Fill the condenser with dry ice, then add acetone slowly until the cold finger is about one third full.
- 11.7.1.4. Mix 10.0 g diazald and 90 mL ether. Place in a separatory funnel over the reaction vessel.
- 11.7.1.5. 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.
- 11.7.1.6. When the diazald solution has been used up, slowly add 10 mL of ether and continue distillation until the distillate is colorless.
- 11.7.1.7. 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.
- 11.7.2. Disposal of outdated diazomethane solutions
 - 11.7.2.1. Add 20 mL of ether to 2 mL of glacial acetic acid in a large beaker in the hood.
 - 11.7.2.2. 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.
 - 11.7.2.3. Dispose of the ether solution in the non-chlorinated wastes bottle.
- 11.8. Esterification (Diazomethane solution method)
 - 11.8.1. Add approximately 2 mL of diazomethane solution and let sit for 10-15 minutes.
 - 11.8.2. Add approximately 0.2 g of silicic acid to the extract. Allow to spontaneously evaporate to about 1.0 mL, then make up to 10 mL with hexane.
 - 11.8.3. Extract is ready for analysis by gas chromatography.

11.9. Esterification (Bubbler Method)

- 11.9.1. Assemble the diazomethane apparatus (Figure A1) 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.
- 11.9.2. Place the tip of the disposable pipet 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 pipet 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).
- 11.9.3. 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.
- 11.9.4. 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 II, September 1994, Chlorinated Herbicides, Method 8150B.

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. Directions to add sodium sulfate to the soil sample until a free flowing texture is achieved have been added. In common with other SW-846 extraction procedures, this improves the extraction efficiency. (Section 11.1.4)
- 17.1.3. For the soil extraction. the acetone and ether are added together rather than separately. (Section 11.1.6)
- 17.1.4. The requirement for the sulfuric acid added in the solvent extraction to be cold has been removed. Since a small quantity of acid is added to a large quantity of extract. nothing is gained by having the acid cold. (Section 11.5.1)
- 17.1.5. Silicic acid is stored at room temperature.
- 17.1.6. 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

In the hydrolysis procedure. the extract is added to the potassium hydroxide solution rather than visa versa.

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

¹The 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
	Dalapon		8
	2,4,5-T		4

¹The herbicide spiking solution contains the herbicides as the free acids.

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 8151.

2. SUMMARY OF METHOD

This method is based on SW846 method 8151. 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
- 5.4. Diethyl ether must be free of peroxides as demonstrated by EM Quant test strips.
- 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.
 - 7.2.5. Diazald. 99% purity
 - 7.2.6. 2,3,4,5,6-Pentafluorobenzyl bromide (PFBBBr) $C_6H_4CH_2Br$
 - 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 slurring 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 H₂SO₄. (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 mL of herbicide matrix spiking solution. (Refer to tables B1 and B2)
- 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 esters are to be determined. This is normally the case. If the herbicide esters are not to be determined, omit this step and go to 11.1.4.
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 ≥ 12 adjust to ≥ 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 (and 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 vigorously for 2 minutes. (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.
- 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 .
 - 11.1.11. Add 120 mL diethyl ether to the sample and extract by shaking vigorously for 2 minutes. (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 sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of 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 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 mL of herbicide matrix spiking solution. (Refer to tables B1 and B2)
- 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%.
- 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 anhydrous 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 10g of anhydrous 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 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 ≥ 12 , adjust with additional KOH.

11.2.16. Heat on a water bath at 60-60°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.** The aqueous solution contains the herbicides.

11.2.18. Adjust the pH of the solution to ≤ 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. (See section 11.2.14) 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 section 11.2.12 or 11.2.20, using a small separatory funnel. **Discard the organic phase.**

11.3.2. Adjust the pH of the solution to ≤ 2 with cold (4°C) sulfuric acid. (1:1).
Extract once with 40 mL diethyl ether and twice with 20 mL diethyl ether.

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 sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of 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, 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. Carefully disassemble the concentrator tube and rinse the lower glass joint with a small amount of diethyl ether.

11.5.4. Add 0.1 mL of methanol.

11.5.5. 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 2 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 10 mL with hexane.

11.6.3.3. Extract is ready for analysis by gas chromatography.

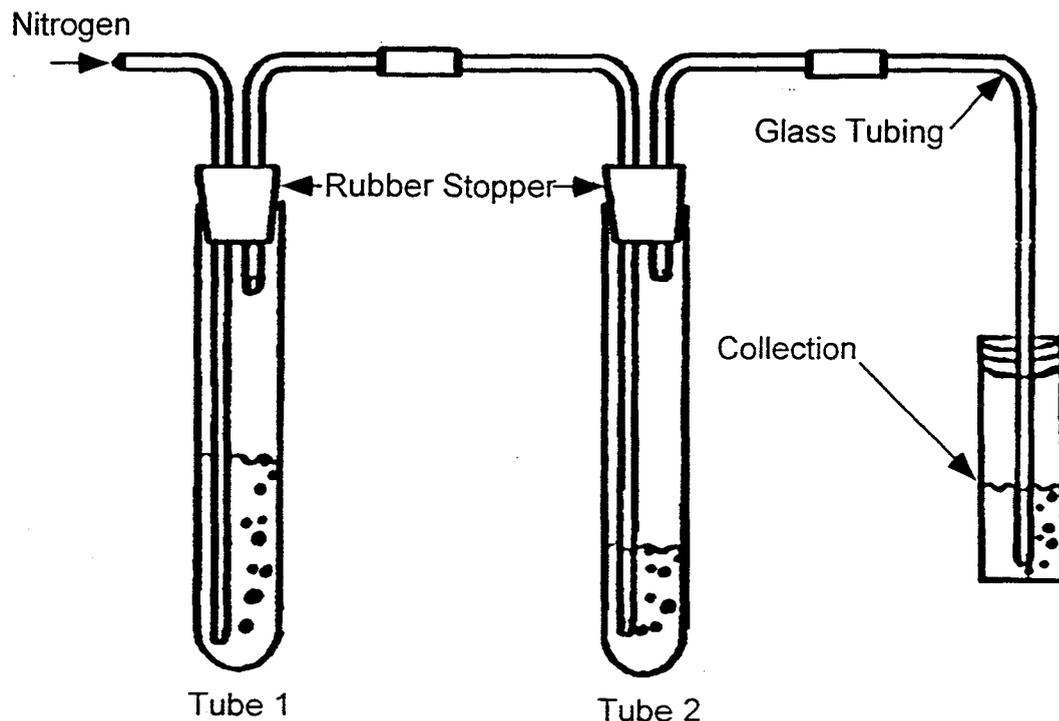
11.7. Esterification (Bubbler Method)

11.7.1. Assemble the diazomethane apparatus (Figure A1) 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 pipet 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 pipet 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 II. September 1994. Chlorinated Herbicides. Method 8151.

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

None

17.3. Tables

Table B1 Herbicide Surrogate Spiking Solutions		
Analyte Group	Surrogate Spike Solution ID	Volume (mL)
Herbicides	Herbicides SS	1.0

Table B2 Herbicide Matrix Spike and LCS Solutions		
Analyte Group	Matrix Spike Solution ID	Volume (mL)
Herbicides	Herbicides MS	1

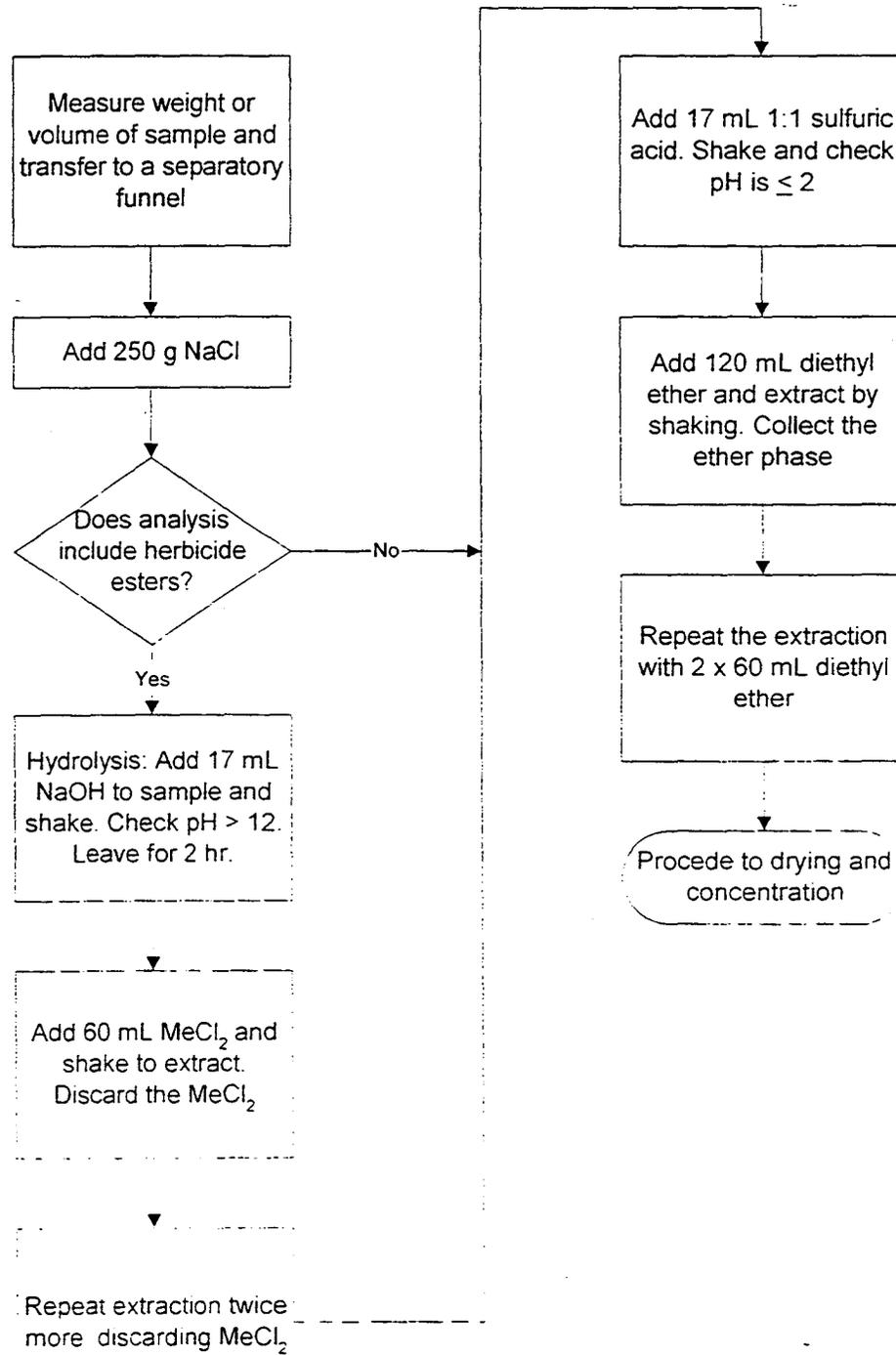
Table B3 Herbicide Surrogate Spike Components			
Type	Compounds ¹	Solvent	Conc. (ug/mL)
Herbicides SS	2,4-DCAA	Methanol	16

¹The surrogate is spiked as the free acid

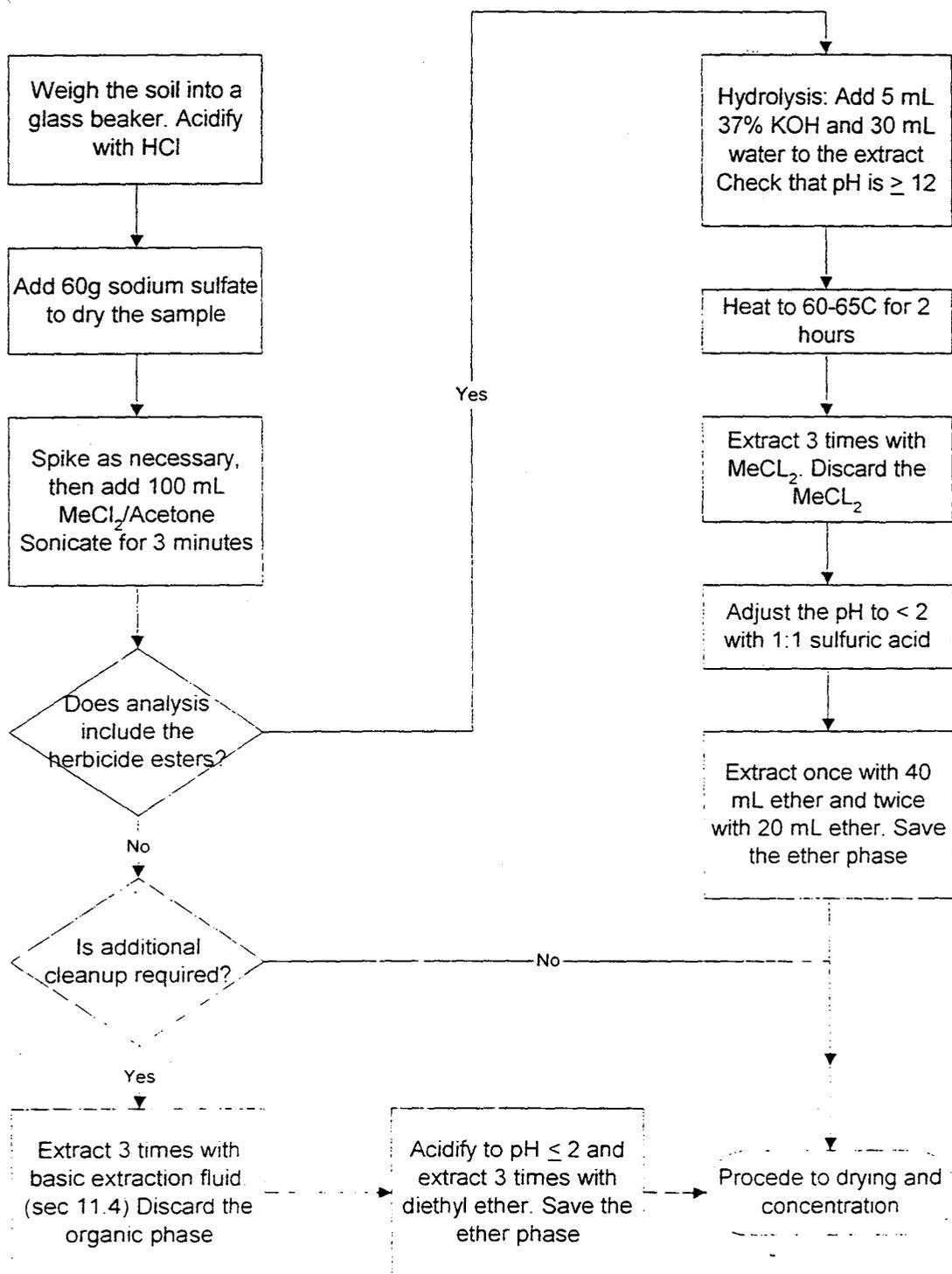
Table B4			
Herbicide Matrix Spike Components			
Type	Compounds ¹	Solvent	Conc. (ug/mL)
Herbicides MS	2,4-D	Methanol	16
	2,4-DB		16
	2,4,5-TP (Silvex)		4
	Dalapon		8
	2,4,5-T		4

¹The herbicide spiking solution contains the herbicides as the free acids.

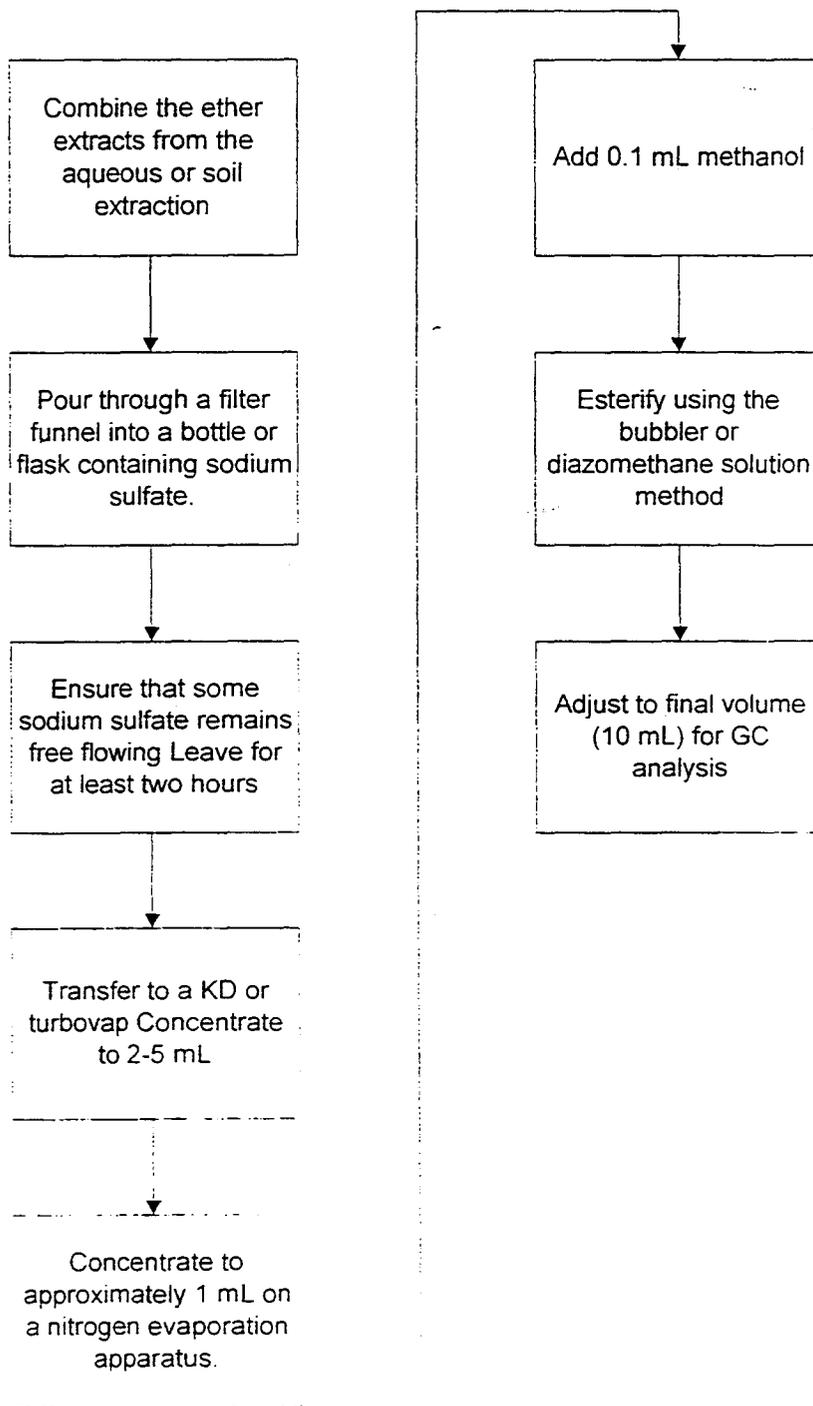
Extraction of Aqueous Samples



Extraction of Soils and Sediments



Drying, Concentration and Esterification



**APPENDIX B
EXTRACTION PROCEDURE FOR CHLORINATED
ACID HERBICIDES BASED ON METHOD 8151**

SOP No: CORP-OP-0001NC

Revision No: 2.4

Revision Date: 10/16/98

Page 94 of 95 _____

(This page intentionally left blank)

**APPENDIX B
EXTRACTION PROCEDURE FOR CHLORINATED
ACID HERBICIDES BASED ON METHOD 8151**

SOP No: CORP-OP-0001NC

Revision No: 2.4

Revision Date: 10/16/98

Page 95 of 95

(This page intentionally left blank)

APPENDIX C

LABORATORY ANALYSIS CRITERIA

Table 1
Summary of Method Quality Objectives for Method 6010
ICP metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (9.2.1.1)	3-stds and blank	Daily	$r \geq 0.995$
Instrumental Precision (9.2.1.1)	RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	RSD < 5%
Initial Calibration Verification (ICV) (9.3)	1. Mid-level (2nd source) verification 2. QC/MRL : Low-level Check standard at MRL	After initial calibration	QC limits = 90-110% QC limits = 80-120%
Initial Calibration Blank (ICB) (9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL Check Sample (-2X MDL)
Interelement Check Standards (ICS) (8.1)	ICS-A - interferents only ICS-B - interferents and target analytes	Beginning of analytical sequence	QC limits = 80-120% for target analytes
Continuing Calibration Blank (CCB) (9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL Check Sample (-2X MDL)
Continuing Calibration Verification (CCV) (9.5 / 9.5.1)	1. Mid-level verification 2. QC/MRL	Every 10 samples and at end of analytical sequence	QC limits = 90-110% QC limits = 80-120%
Method Blank (MB) (10.2.1 / 11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < MDL Check Sample (-2X MDL)
Laboratory Control Sample (LCS) (10.2.2 / 11.4.2)	Interference-free matrix containing all target analytes	1 per sample batch	Recovery = 80 - 120%
Matrix Spike (MS) (10.2.3 / 11.4.3 11.4.3.1)	Sample matrix spiked with all/subset of target analytes prior to digestion	1 per sample batch	Recovery = 75 - 125%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (10.2.4 / 11.4.4)	Refer to text for MD or Ms.	1 per sample batch	RPD ≤ 25

Table 1 (Continued)
 Summary of Method Quality Objectives for Method 6010
 ICP metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Post Digestion Spike (PDS) (10.3.1 / 11.4.6)	Sample digestate spiked with all/subset of target analytes	As needed to confirm matrix effects	Recovery = 75 - 125%
Serial Dilution (SD) (10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results V 10%
Method of Standard Addition (MSA) (12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	$r \geq 0.995$

1 The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if between seven (7) to fifteen (15) metals are reported from the ICP analysis, one (1) SMF is allowed to the expanded criteria presented. If greater than 15 metals are reported from the ICP analysis, two (2) SMFs are allowed. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

Table 2
Summary of Method Quality Objectives for Method 7000 series
GFAA/CVAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (9.2.1.2)	3 stds and blank	Daily	$r \geq 0.995$
Instrumental Precision (9.2.1.2)	RPD of 2 injections	All standards, and ICV/CCV	RPD ≤ 10
Initial Calibration Verification (ICV) (9.3)	1. Mid-level (2nd source) Verification 2. QC/MRL: Low level std	After initial calibration	QC limits = 90-110% QC limits: 80-120%
Initial Calibration Blank (I CB) (9.4)	Interference-free matrix To assess analysis contamination	After initial calibration	Analytes < MDL Check Sample (-2X MDL)
Continuing Calibration Blank (CCB) (9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL Check Sample (-2X MDL)
Continuing Calibration Verification (CCV) (9.5 / 9.5.1)	1. Mid-level verification 2. QC/MRL	Every 10 samples and at end of analytical sequence	QC limits = 80-120% QC limits=80-120%
Method Blank (MB) (10.2.1 / 11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < MDL Check Sample (-2X MDL)
Laboratory Control Sample (LCS) (10.2.2 / 11.4.2)	Interference-free matrix containing target analytes	1 per sample batch	%Rec = 80% - 120%
Matrix Spike (MS) (10.2.3 / 11.4.3/ 11.4.3.1)	Sample matrix spiked with target analytes prior to digestion	1 per sample batch	%Rec = 80% - 120%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (10.2.4 / 11.4.4)	Refer to text for MID or Ms.	1 per sample batch	RPD ≤ 20
Post Digestion Spike (PDS) (10.3.1 / 11.4.6)	Sample digestate spiked with target analytes	As needed to confirm matrix effects	Recovery = 85 - 115%
Serial Dilution (SD) (10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results $\geq 10\%$
*Method of Standard Addition (MSA) (12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	$r \geq 0.995$

Table 3
Summary of Method Quality Objectives for Method 8021
VOCS

QC Element	Target Analyte / Surrogate	Poor Purgers / Gases / Sporadic Marginal Failures ¹
Initial Calibration (9.2.2.1)	Primary Evaluation: $r \geq 0.995$, $RSD \leq 20\%$, $r^2 \geq 0.990$	No allowance
ICV (9.3)	1. Recovery = 85 - 115% 2. QC/MRL: $D \leq 15\%$	Sporadic Marginal Failures: Recovery = 70 - 130%
CCV (9.5 / 9.5.2 9.5.2.1)	1. Primary Evaluation: Drift $\leq 15\%$, $D \leq 15\%$ Alternative Evaluation: Mean Drift/D For all target analytes $\leq 15\%$ 2. QC/MRL: $D \leq 15\%$	Primary Evaluation: Drift $\leq 20\%$, $D \leq 20\%$ Alternative Evaluation: Maximum allowable Drift/D For each target analyte $\leq 30\%$
MB (10.2.1 / 11.4.1)	Target Analytes: Analytes < MDL Check Sample (-2X MDL)	Common Lab Contaminants: Analytes < MRL
LCS (10.2.2 / 11.4.2)	Water: Recovery, 80 - 120% Soil: Recovery, 75 - 125%	Sporadic Marginal Failures: Recovery = 60 - 140%
IVIS (10.2.3/ 11.4.3/ 11.4.3.2)	Recovery = 70 - 130%	Sporadic Marginal Failures: %Rec = 60% - 140%
MSD/MD (10.2.4 / 11.4.4)	Water: RPD ≤ 30 Soil: No RPD Limits	Water: RPD ≤ 40 Soil: No RPD Limits
Surrogates (10.2.5 11.4.5)	Interference-Free Matrix: Water: Recovery 80 - 120% Soil: Recovery 75 - 125% Project Sample Matrix: Recovery = 70 - 130%	Not Applicable
Target Analyte Confirmation (12.3)	RPD ≤ 40	RPD ≤ 40

¹ The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if the 8020 Target Analyte List (10 compounds) is reported, 1 SMF is allowed. If the 8010 Target Analyte List (32 compounds) is reported, 3 SMFs are allowed. If the full 8021 Target Analyte List (60 compounds) is reported, 4 SMFs are allowed. If the MS includes only a subset of compounds, allow only one (1) SMF for that QC element. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

Table 4
Summary of Method Quality Objectives for Method 8081
Pesticides

QC Element	Target Analyte/Surrogate	Sporadic Marginal Failure
DDT/Endrin %Breakdown (8.2)	DDT & Endrin Breakdown $\leq 15\%$ each	Not Applicable
Initial Calibration (9.2.2.2)	<u>Primary Evaluation:</u> $r \geq 0.995$, $RSD \leq 20\%$, $r^2 \geq 0.990$ <u>Alternative Evaluation:</u> Mean RSD for all target analytes $\leq 20\%$	No allowance <u>Alternative Evaluation:</u> Maximum allowable RSD for each target analyte $\leq 40\%$
ICV (9-3 / 9.3.1)	1. Recovery = 85 - 115% 2. <u>QC/MRL:</u> Recovery 85-115%	<u>Sporadic Marginal Failures':</u> Recovery = 70 - 130%
CCV (9.5 / 9.5.2 / 9.5.2.2)	1. <u>Primary Evaluation:</u> Drift $\leq 15\%$, $D \leq 15\%$ <u>Alternative Evaluation:</u> Mean Drift (D) for all target analytes $\leq 15\%$ 2. <u>QC/MRL:</u> $D \leq 15\%$	No allowance <u>Alternative Evaluation:</u> Maximum allowable Drift, D for each target analyte $\leq 30\%$
MB (10.2.1 / 11.4.1)	Analytes < MDL Check Sample (-2X MDL)	Not Applicable
LCS (10.2.2 / 11.4.2)	<u>Water:</u> Recovery = 50 - 130% <u>Soil:</u> %Recovery = 50 - 130%	<u>Sporadic Marginal Failures':</u> Recovery = 30-150%
MS (10.2.3 / 11.4.3 / 11.4.3.2)	Recovery = 40 - 140%	<u>Sporadic Marginal Failures':</u> Recovery = 30 - 150%
MSD/MD (10.2.4 / 11.4.4)	RPD ≤ 50	RPD ≤ 60
Surrogates (10.2.5 / 11.4.5)	<u>Interference- Free Matrix:</u> <u>Water:</u> Recovery = 50 - 130% <u>Soil:</u> Recovery = 50 - 130% <u>Project Sample Matrix:</u> Recovery = 40 - 140%	Not Applicable
Target Analyte Confirmation (12.3)	RPD ≤ 40	RPD ≤ 40

The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if the full list of 21 compounds are reported from the GC/ECD analysis, then two (2) SMFs are allowed to the expanded criteria presented. If the MS includes only a subset of compounds, allow only one (1) SMF for that QC element. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

Table 5
Summary of Method Quality Objectives for Method 8082
PCBs

QC Element	Target Analyte/Surrogate
Initial Calibration (9.2.2.3)	$r \geq 0.995$, $RSD \leq 20\%$, $r^2 \geq 0.990$
ICV (9.3 / 9.3.2)	1. Recovery = 85 - 115% 2. QC/MRL: recovery = 85-115%
CCV (9.5 / 9.5.2)	1. Drift $\leq 15\%$, $D \leq 15\%$ 2. QC/MRL: $D \leq 15\%$
M B (10.2.1 / 11.4. 1)	Analytes < IVIDL Check Sample (-2X IVIDL)
LCS (10.2.2 1 11.4.2)	<u>Water</u> : Recovery = 50 - 130% <u>Soil</u> : Recovery = 50 - 130%
M S (10. 2.3 / 11.4.3)	Recovery = 40 - 140%
MSD/MD (10.2.4/ 11.4.4)	RPD ≤ 50
Surrogates (10.2.5 /11.4.5)	<u>Interference- Free Matrix</u> : Water: Recovery = 50 - 130% Soil: Recovery = 50 - 130% <u>Project Sample Matrix</u> : Recovery = 40 - 140%
Target Analyte Confirmation (12.3)	RPD ≤ 40

Table 7
Summary of Method Quality Objectives for Method 8270 Semivolatiles

QC Element	Target Analyte/Surrogate	Poor Performers/ Sporadic Marginal Failures'
Initial Calibration (9.2.2.5)	<u>Instrument Evaluation:</u> SPCCs: minimum RF values per method Requirements CCCs: verify RSD \leq 30% <u>Primary Evaluation (all target analytes)</u> $r \geq 0.995$, RSD \leq 15%, $r^2 \geq 0.990$ <u>Alternative Evaluation.</u> Mean RSD for all target analytes \leq 15%	No allowance <u>Alternative Evaluation:</u> Maximum allowable RSD for each target analyte $<$ 40%
ICV (9.3)	1. Recovery = 70 - 130% 2. QC/MRL: D $<$ 20%	<u>Sporadic Marginal Failures':</u> Recovery = 50 - 150%
CCV (9.5 / 9.5.2 9.5.2.4)	<u>Instrument Evaluation:</u> 1. SPCCs: minimum RF values per method requirements 2. CCCs: verify D \leq 30% 3. Primary Evaluation (CCCs) Drift \leq 20%, D \leq 20% 4. QC/MRL: D \leq 20%	<u>Primary Evaluation (remaining target analytes):</u> Qualitative, see text
MB (10.2.1 / 11.4.1)	<u>Target Analytes:</u> Analytes $<$ MDL Check Sample (-2X MDL)	<u>Common Lab Contaminants:</u> Analytes \leq MDLs
LCS (10.2.2 / 11.4.2)	<u>Water:</u> Recovery = 60 - 120% (-15 analytes) = 45 - 135% (-30 analytes) = 20 - 150% (-45 analytes) <u>Soil:</u> Recovery = 60 - 120% (-20 analytes) = 45 - 135% (-25 analytes) = 30 - 150% (-45 analytes)	<u>Sporadic Marginal Failures':</u> <u>Water:</u> Recovery = 15 - 150% <u>Soil:</u> Recovery = 25 - 150%
MS (10.2.3 11.4.3 11.4.3.2)	<u>Water:</u> Recovery = 45 - 135% <u>Soil:</u> Recovery = 45% - 135%	<u>Sporadic Marginal Failures':</u> <u>Water:</u> Recovery = 15% - 150% <u>Soil:</u> Recovery = 20% - 150%
MSD/MD (10.2.4 / 11.4.4)	<u>Water:</u> RPD \leq 50 <u>Soil:</u> RPD \leq 60	<u>Sporadic Marginal Failures':</u> <u>Water:</u> RPD \leq 60 , <u>Soil:</u> RPD \leq 60
Surrogates (10.2.5 / 11.4.5)	<u>Interference- Free Matrix:</u> <u>Water:</u> Recovery = 60 - 120% B/N cmpds Recovery = 45 - 135% A cmpds <u>Soil:</u> Recovery = 60 - 120% B/N cmpds Recovery = 45 - 135% A cmpds <u>Project Sample Matrix:</u> <u>Water:</u> Recovery = 45 - 135% B/N cmpds Recovery = 35 - 140% A cmpds <u>Soil:</u> Recovery = 45 - 135% B/N cmpds Recovery = 35 - 140% A cmpds	<u>Sporadic Marginal Failures':</u> <u>Water:</u> Recovery = 15 - 150% <u>Soil:</u> Recovery = 20 - 150%

Table 8
Summary of Method Quality Objectives for Method 8330
Explosives

QC Element	Target Analyte/Surrogate	Tetryl / Sporadic Marginal Failures ¹
Initial Calibration (9.2.2.6)	<u>Primary Evaluation:</u> $r \geq 0.995$, $RSD \leq 20\%$, $r^2 \geq 0.990$ <u>Alternative Evaluation:</u> Mean RSD for all target analytes $\leq 20\%$	No allowance <u>Alternative Evaluation:</u> Maximum allowable RSD for each target analyte $\leq 40\%$
ICV (9.3)	1. Recovery = 85 - 115% 2. <u>QC/MRL: D</u> $\leq 15\%$	<u>Sporadic Marginal Failures¹:</u> Recovery = 70 - 130%
CCV (9.5 / 9.5.2)	1. <u>Primary Evaluation:</u> Drift $\leq 15\%$, $D \leq 15\%$ <u>Alternative Evaluation:</u> Mean Drift (D) for all target analytes $\leq 15\%$ 2. <u>QC/MRL: D</u> $\leq 15\%$	<u>Primary Evaluation:</u> Drift $\leq 20\%$, $D \leq 20\%$ <u>Alternative Evaluation:</u> Maximum allowable Drift (D) for each target analyte $\leq 30\%$
MB (10.2.1 / 11.4.1)	<u>Target Analytes:</u> Analytes < MDL Check Sample (-2X MDL)	Not Applicable
LCS (10.2.2/11.4.2)	<u>Water:</u> Recovery = 60 - 120% <u>Soil:</u> Recovery = 60 - 120%	<u>Sporadic Marginal Failures¹:</u> Recovery = 40 - 150%
MS (10.2.3 / 11.4.3/11.4.3.2)	Recovery = 50 - 140%	<u>Sporadic Marginal Failures¹:</u> Recovery = 40 - 150%
MSD/M D (10.2.4 11.4.4)	RPD ≤ 50	RPD ≤ 60
Surrogates (10.2.5 / 11.4.5)	<u>Interference-Free Matrix:</u> <u>Water:</u> Recovery = 60 - 140% <u>Soil:</u> Recovery = 50 - 150% <u>Project Sample Matrix:</u> Recovery = 50 - 150%	Not Applicable
Target Analyte Confirmation (12.3)	RPD ≤ 40	RPD ≤ 40

¹ The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if between seven (7) to fifteen (15) explosives are reported from the HPLC analysis, one (1) SMF is allowed to the expanded criteria presented for the ICV and LCS. If greater than 15 explosives are reported, two (2) SMFs are allowed for the ICV and LCS. If the MS includes only a subset of compounds, allow only one (1) SMF for this QC element. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

² Due to the tendency for Tetryl to decompose, an expanded criteria may be applied at 45% - 140% for both water and soil matrices.

APPENDIX D

DATA VALIDATION CHECKLIST

VOLITILE ORGANIC DATA VALIDATION CHECKLIST

Laboratory _____

SDG Number _____

Section I-Part A is filled out if the data package contains VOA analyses.

The review consisted of checking and verifying that the following performance criteria are within acceptable QC limits.

- x Holding Time Review
- x System Monitoring Compound (Surrogate) Review
- x Matrix Spike/Matrix Spike Duplicate Review
- x Blank Contamination Review
- x GC/MS Instrument Performance Check
- x Initial and Continuing Calibration Check Internal Standard Areas Check

Any exceedance in the QC limits are documented in the attached summary sheets. Please note that field/rinse blanks, holding times for non-detects, as well as qualifying detected values below the CRQL will be analyzed as a separate process.

The following qualifiers may be applied:

J = Positive result at an estimated value

R = Data is unreliable due to significant QC problems

B = Blank contamination

Data Reviewer _____

VOA ANALYSES1. Chain-of-Custody Records and Requests for Analyses (C-O-C/RFA)

Are C-O-C/RFA Records present for all samples?

Yes No N/A

Do the C-O-C/Request for Analysis or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems, or special circumstances affecting the quality of the data?

Yes No N/A

Action: Use professional judgment to evaluate the effect on the quality of the data.

Note: Holding times information will be available from the IRDMIS database. Only verification will be performed during validation.

2. System Monitoring Compound (Surrogates) Analysis

Are all system monitoring compound recoveries below the upper acceptance limit (UJL)?

Yes No N/A

Action: If a system monitoring compound recovery is above the upper acceptance level (UL) qualify all associated positive values as estimated "J".

Are all system monitoring compound recoveries above the lower acceptance level (LL)?

Yes No N/A

Action: If a system monitoring compound recovery is below the lower acceptance limit (LL) qualify all associated detected data estimated "J".

Note: Professional judgment should be used to qualify data that have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard areas.

Common Lab Contaminants - Methylene Chloride, Acetone, Toluene, 2-butanone, Carbon Disulfide

Note: Trip blanks are used to qualify only those samples with which they were shipped and are only required for VOA matrices. Blanks may not be qualified because of contamination in another blank. Field Blanks and Trip Blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems. Field blank contamination is not to be applied at this time. This will be done as a separate step in the validation process. If all associated samples for the Trip Blanks are not found in the package please note the exceptions on the "TRIP BLANK TO BE VALIDATED LIST". Do not apply qualifiers to rinse blanks, (which are denoted as R~SW under Site Type on the COC Form).

Has an instrument performance compound been analyzed for every 12 hours of sample analysis per instrument. Yes No N/A

Action: Reject (R) all data generated outside an acceptable 12-hour tune interval.

Have the ion abundance's been normalized to m/z - 9j? Yes No N/A

Action: If mass assignment is in error, qualify all associated data as unusable (R).

7. GC/TV~S Initial Calibration

Are the Initial Calibration Forms present and complete for the volatile fraction? Yes No N/A

Action: If any calibration standard forms are missing, please contact lab.

Are the RRFs above 0.05 for Target/HSL compounds? Yes No N/A

Action: If the RRF is below the above listed criteria qualify all associated analyze detected values estimated "J" and rejects "R" all associated analyze non-detects.

Are any % RSD's above 30% for any sample analyze?

Yes No N/A

Action: If %RSD >30% qualify all associated detected sample analyses estimated "J".

8. Continuing Calibration

Are all continuing calibration forms present and complete for the volatile fraction?

Yes No N/A

Action: If any continuing calibration forms are missing or incomplete, please notify laboratory.

Are all RRFs above 0.05 for Target/HCL compounds?

Yes No N/A

Action: If the RRF is below the above mentioned criteria qualify all detected values as estimated "J" and all non-detects as rejected "R".

Are any % D's above 25% for any volatile analyze?

Yes No N/A

Action: If any %D exceeds 25% qualify all associated detected sample analyses as estimated "J"

9. Internal Standard

Are the internal standard areas of every sample and blank within the upper and lower limits for each continuing calibration?

Yes No N/A

Action: If the internal standard area count is outside the upper or lower limit, flag "J" all associated positive results. If extremely low area counts (~ 25%) are reported, or if performance exhibits a major abrupt drop-off, qualify all associated non-detects as "R", rejected and all associated detects as estimated, "J".

Note: If the IS area is above 150%, use professional judgment.

Are the retention times of the internal standards within 30 seconds of the associated calibration standard? Yes No N/A

Action: Professional judgment should be used to qualify data if the retention times differ by more than 30 seconds to determine if a-false positive or negative exists.

APPENDIX B**BASE/NEUTRAL/ACID (BNA)
ORGANIC DATA VALIDATION CHECKLIST**

BNA ORGANIC DATA VALIDATION CHECKLIST

Laboratory _____

SDG Number _____

Appendix C is filled out if the data package contains B~A analyses.

The review consisted of checking and verifying that the following performance criteria are within acceptable QC limits.

- x Holding Time Review
- x System Monitoring Compound (Surrogate) Review
- x Matrix Spike/Matrix Spike Duplicate Review
- x Blank Contamination Review x GC/MS Instrument Performance Check
- x Initial and Continuing Calibration Check Internal Standard Areas Check

Any exceedance in the QC limits are documented in the attached summary sheets. Please note that field/rinse blanks, holding times for non-detects, as well as qualifying detected values below the CRQL will be analyzed as a separate process.

The following qualifiers may be applied:

J = Positive result at an estimated value

R = Data is unreliable due to significant QC problems

B = Blank contamination

Data Reviewer _____

1. Chain-of-Custody Records and Request for Analysis (C-O-C/RFA)

Are C-O-CIRFA Records present for all samples?

 Yes No N/A

Action: If no, note on the request from the laboratory.

Do the C-O-C/RFA or lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems, or special circumstances affecting the quality of the data?

 Yes No N/A

Action: Use professional judgment to evaluate the effect on the quality of the data.

Note: Holding times will be reviewed.2. System Monitoring Compound (Surrogates) Analysis

Were two or more base-neutral OR acid SMCs out of specifications for any sample or method blank?

 Yes No N/A

If yes, were samples reanalyzed?

 Yes No N/ANote The theoretical value for the surrogates was 50 ug/ml for base/neutrals and 100 ug/ml for acids.Action: If all BNA system monitoring compounds (SMC) recoveries are >10% but two within the base-neutral or acid fraction do not meet specifications, for the affected fraction only (i.e., base-neutral or acid compounds, flag all detected results as estimated (J).

Were any base-neutral ~2B acid SMC's below 10% for any sample?

Yes No N/A

Action: If any SMC's are below 10% qualify all detects estimated "J" and reject "R" all non-detects for the affected fraction (i.e. Base-neutral or acid).

Note: Professional judgment should be used to qualify data that have method blank system monitoring compound recoveries out of specification in both original and reanalysis. Check the internal standard areas.

3. Matrix Spikes

Were any matrix spikes analyzed?

a. Water Yes No N/A
b. Soil Yes No N/A

Action: If any matrix spike data are missing, note in the case narrative.

4. Blanks

Has a reagent/method blank analysis been reported per set of samples of similar matrix, or concentration level, and for each extraction batch? Yes No N/A

Action: If the above information is missing, please notify the laboratory.

5. Contamination

Do any method/instrument/reagent blanks have positive results (Target and/or TIC) for BNAs? when necessary. Yes No N/A

Action: For common lab contaminants, Sample concentrations less than ten times and all other sample analyses sample concentrations less than five times the blank value qualify "B" in both the USAT and EPA qualification fields. Use the largest value from all the associated blanks. Do not apply method blank contamination to rinse blank samples.

Common Contaminants - Common phthalate esters

Note: Field blank contamination is not to be evaluated at this time. This will be done as a separate step in the validation process. Do not apply method blank contamination to rinse blank samples.

6. GC/IVIS Instrument Performance Check

Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each 12-hour shift?

Action: If the above information is missing, please notify the laboratory.

Has an instrument performance compound been analyzed for every 12 hours of sample analysis per instrument? Yes No N/A

Action: If no, use professional judgment to qualify the data

Have the ion abundance's been normalized to m/z 198? Yes No N/A

Action: If mass assignment is in error, flag all associated sample data as unusable (R).

Have all ion abundance criteria been met? Yes No N/A

Action: If ion abundance criteria are not met, use professional judgment to determine what action, if any, is required.

7. GC/MS Initial Calibration

Are the Initial Calibration Forms present and complete for the TCL/BNA fraction?

Yes No N/A

Action: If any calibration standard forms are missing, please contact lab.

Are all RRF's above 0.05 for Target/HSL compounds?

Yes No N/A

Action: If the RRF is below the above listed criteria qualify all associated analyze detected values contaminated "J" and reject "R" all associated non-detects.

Are any % RSD's above 30% for any sample analyze?

Yes No N/A

Action: If %RSD >30% qualify all associated detected sample analyses estimated "J".

8. GC/MS Continuing Calibration

Are the Continuing/Weekly Calibration Forms present and complete for the BNA fraction (TCL + extra compounds)?

Yes No N/A

Has a continuing calibration standard been analyzed for every 12 hours of sample analysis/instrument?

Yes No N/A

Action: If any forms are missing or no continuing calibration standard has been analyzed within 12 hours of every sample analysis, please request from laboratory. If continuing calibration data are not available and were required, flag all associated sample data as unusable (R).

Are all Continuing RRF's above 0.05 for Target/HCL compounds?

Yes No

N/A

Action: If any RRF is below the above mentioned criteria qualify all detected values as estimated "J" and all non-detects as rejected "R".

Are any % D's above 25% for any semi-volatile analyze?

Yes No

N/A

Action: If any % D exceeds 25% qualify all associated detected sample analyses as estimated "J".

9. Internal Standard

Are the internal standard areas of every sample and blank with the upper and lower limits for each continuing calibration?

Yes No

N/A

Action: If the internal standard area count is outside the upper or lower limit, flag "J" all associated positive results. If extremely low area counts (< 25 %) are reported, or if performance exhibits a major abrupt drop-off, qualify all associated non-detects as "R", reject and all associated detects as "J", estimated.

Note: If the IS area is above 150%, use professional judgment.

Are the retention times of the internal standards within 30 seconds of the associated calibration standard?

Yes No

N/A

Action: Professional judgment should be used to qualify data if the retention times differ by more than 30 seconds to determine if a false positive or negative exists.

PESTICIDE/PCB DATA VALIDATION CHECKLIST

Laboratory

SDD Number

Appendix C is filed with the data package covering field work only.

The review consisted of checking and verifying that the following performance criteria are satisfied for each QC blank:

APPENDIX C

PESTICIDE/PCB DATA VALIDATION CHECKLIST

- x Holding Time Review
- x Matrix Spike/Recovery Data Comparison Review
- x Blank Contamination Review
- x GCM Performance Review
- x Control and Outlying Values in Check Standard Results (Yes/No)

All out of the upper limit for the QC limits are documented in the attached recovery sheet. Please note that field team blanks, including those for recoveries, as well as duplicate samples, values below the GRL will be analyzed in a separate review.

The following guidelines may be applied:
1 - Positive result is an estimated value
2 - Data is available due to significant QC problems
3 - Blank contamination

Data Reviewer

PESTICIDES/PCB DATA VALIDATION CHECKLIST

Laboratory _____

SDG Number _____

Appendix D is filled out if the data package contains Pesticide/PCB analyses.

The review consisted of checking and verifying that the following performance criteria are within acceptable QC limits.

- x Holding Time Review
- x System Monitoring Compound (Surrogate) Review
- x Matrix Spike/Matrix Spike Duplicate Review
- x Blank Contamination Review
- x GC/MS Instrument Performance Check
- x Initial and Continuing Calibration Check Internal Standard Areas Check

Any out of the upper limit in the QC limits are documented in the attached summary sheets. Please note that field/rinse blanks, holding times for non-detects, as well as qualifying detected values below the CRQL will be analyzed as a separate process.

The following qualifiers may be applied:

J = Positive result at an estimated value

R = Data is unreliable due to significant QC problems

B = Blank contamination

Data Reviewer _____

PESTICIDE/PCB ANALYSIS

1. Chain-of Custody Records and Requests for Analysis (C-O-C/RFA)

Are C-O-C/Request For Analysis Records present for all samples?

Yes No N/A

Action: If no, please contact laboratory.

Do the C-O-C/Request For Analysis or Lab narrative indicate any problems with sample receipt, condition of samples, analytical problems, or special circumstances affecting the quality of the data?

Yes No N/A

Action: Use professional judgment to evaluate the effect on the quality of the data.

Note: Holding times will be reviewed using the I-DMIS system.

2. System Monitoring Compound (Surrogate) Recovery

Were System Monitoring Compound recoveries of DBC outside of the contract specification (20-150) for any sample or blank?

Yes No N/A

Action: No qualification is done if system monitoring compounds (SMC) are diluted out. If recovery for SMC is below the contract limit, but above 10%, flag positive results for that sample as "J". If recovery is < 10%, qualify positive results as "J" and reject "R" non-detects. If recovery is above the contract advisory limits, qualify positive values as "J".

Are all surrogates Retention Times within +/- 0.10 minutes for DBC in the calibration curve?

Yes No N/A

Action: If the Retention time limits are not met, the analysis may be qualified unusable "R" for that sample on the basis of professional judgment.

3. Matrix spikes

Were matrix spikes analyzed?

- | | | | |
|----------|------------------------------|-----------------------------|------------------------------|
| a. Soil | <input type="checkbox"/> Yes | <input type="checkbox"/> No | <input type="checkbox"/> N/A |
| b. Water | <input type="checkbox"/> Yes | <input type="checkbox"/> No | <input type="checkbox"/> N/A |

Action: If any matrix spike data are missing, note in the case narrative.

4. Blanks

Frequency of Analysis: For the analysis of Pest/PCB TCL compounds, has a reagent/method blank been analyzed for each Sample Delivery Group for each of the matrix or concentration or each extraction batch, whichever is more frequent (Check the prep sheets)?

- Yes No N/A

Action-If not present note in the case narrative.

5. Contamination

Do any method/instrument/reagent blanks have positive results for Pest/PCBs?

- Yes No N/A

Action: Sample concentrations less than five times the blank value qualify "B" in both the USAT and EPA qualification fields. Use the largest value from the associated blanks.

Note: If gross blank contamination exists (i.e., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R) due to interference. Field blank contamination is not to be applied at this time. This will be done as a separate step in the validation process. Do not apply method blank contamination to rinse blank samples.

6. Calibration and GC performance

Is the correlation coefficient of individual analyses in the calibration curve >0.995 ?

Yes No N/A

Action: If no, qualify all associated positive results generated during the entire analytical sequence as estimated (J).

Was Eval B standard analyzed?

Yes No N/A

Has the individual % breakdown exceeded 20.0% on either column? For 4,4'-DDT? For endrin?

No N/A

Yes

Has the combined % breakdown for 4,4'-DDT~Endrin exceeded 30.0% on either column (required in all instances)?

Yes No N/A

Action: If any % breakdown has failed the QC criteria in the initial calibration sequence, qualify all sample analyses in the entire analytical sequence as described below.

Action: If any % breakdown has failed the QC criteria in a EVAL B Verification Calibration, review data beginning with the samples which followed the last in-control standard until the next acceptable PEM and qualify the data as described below.

a. 4,4'-DDT Breakdown: If 4,4'-DDT breakdown is greater than 20.0%: Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT as unusable (R).

ii. Qualify positive results for DDD and/or DDE as preemptively present at an approximated quantify (J).

b. Edrin Breakdown: If edrin breakdown is greater than 20.0%:

Qualify all positive results for endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable (R).

ii. Qualify positive results for endrin ketone and endrin aldehyde as **preemptively** present at an approximated quantity (J).

c. **Combined Breakdown**: If the combined 4,4'-DDT and endrin breakdown is greater than 30.0%: Qualify all positive results for DDT and endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable (R). If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT as unusable (R).

Qualify positive results for endrin ketone and endrin aldehyde as preemptively present at an approximated quantity (J). Qualify positive results for DDD and/or DDE as preemptively present at an approximate quantity (J).

Are the percent difference (%D) values for all EVAL B analyses less than 25%?

Yes No N/A

Action: If no, qualify all associated positive results generated during the analytical sequence as "J".

Have all samples been injected within a 72-hour period beginning with the injection Instrument Blank?

Yes No N/A

Action: If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly.

Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?

Yes No N/A

Action: If retention times are outside established windows in both columns, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results "R".

Are %D values for all verification calibration standard compounds $<25.0\%$?

Yes No N/A

Action: If the %D is $>25.0\%$ for the compound being quantitated, qualify all associated positive results as "J". The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyze which failed the criteria.

7. Pesticide/PCB Identification

Are retention times (RT) of detected compounds within the established RT windows for both columns (quantitation and confirmation)?

Yes No N/A

Action: If the compound is outside the RT windows, adjust the Form I to CRQL as a false positive.

Note. If laboratory did not confirm value on a second, notify the laboratory.

APPENDIX D**NITROAROMATICS & NITRAMINES DATA VALIDATION CHECKLIST
(EXPLOSIVE RESIDUES)**

NITROAROMATICS & NITRAMINES DATA VALIDATION CHECKLIST

Laboratory _____

SDG Number _____

Appendix D is filled out if the data package contains Explosive Residues analyses.

The review consisted of checking and verifying that the following performance criteria are within acceptable QC limits.

- x Holding Time Review
- x System Monitoring Compound (Surrogate) Review
- x Matrix Spike/Matrix Spike Duplicate Review
- x Blank Contamination Review
- x Initial and Continuing Calibration Check Internal Standard Areas/Peaks Check

Any out of the upper limit in the QC limits are documented in the attached summary sheets. Please note that field/rinse blanks, holding times for non-detects, as well as qualifying detected values below the CRQL will be analyzed as a separate process.

The following qualifiers may be applied:

J = Positive result at an estimated value

R = Data is unreliable due to significant QC problems

B = Blank contamination

Data Reviewer _____

NITROAROMATICS & NITRAMINES ANALYSIS

1. Chain-of Custody Records and Requests for Analysis (C-O-C/RFA)

Are C-O-C/Request For Analysis Records present for all samples?

Yes No

N/A

Action: If no, please contact laboratory.

Do the C-O-C/Request For Analysis or Lab narrative indicate any problems with sample receipt, condition of samples, analytical problems, or special circumstances affecting the quality of the data?

Yes No

N/A

Action: Use professional judgment to evaluate the effect on the quality of the data.

Note: Holding times will be reviewed..

2. System Monitoring Compound (Surrogate) Recovery

Were System Monitoring Compound recoveries of surrogate outside of the contract specification for any sample or blank?

Yes No

N/A

Action: No qualification is done if system monitoring compounds (SMC) are diluted out. If recovery for SMC is below the contract limit, reject "R" non-detects. If recovery is above the contract advisory limits, qualify positive values as "J".

Are all surrogates Retention Times within ± 3 SD of the calibration curve?

Yes No

N/A

Action: If the Retention time limits are not met, the analysis may be qualified unusable "R" for that sample on the basis of professional judgment.

3. Matrix spikes

Were matrix spikes analyzed?

a. Soil	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A
b. Water	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A

Action: If any matrix spike data are missing, note in the case narrative.

4. Blanks

Frequency of Analysis: For the analysis of TCL compounds, has a reagent/method blank been analyzed for each Sample Delivery Group for each of the matrix or concentration or each extraction batch, whichever is more frequent (Check the prep sheets)?

Yes No N/A

Action-If not present note in the case narrative.

5. Contamination

Do any method/instrument/reagent blanks have positive results for explosive residues?

Yes No N/A

Action: Sample concentrations less than five times the blank value qualify "B" in both the USAT and EPA qualification fields. Use the largest value from the associated blanks.

Note: If gross blank contamination exists (i.e., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R) due to interference. Field blank contamination is not to be applied at this time. This will be done as a separate step in the validation process. Do not apply method blank contamination to rinse blank samples.

6. Initial Calibration

Is the RSD of the calibration standards for all the points less than 20%?

Yes No N/A

Action: If no, qualify all associated positive results generated during the entire analytical sequence as estimated (J).

7. Continuing Calibration

Is the average response factor within 15%?

Yes No N/A

INORGANIC DATA VALIDATION CHECKLIST

The data provided in this report were obtained from the following sources: _____

The accuracy of the data is based on the accuracy of the original data provided by the sources.

APPENDIX E

INORGANIC DATA VALIDATION CHECKLIST

1 - Title page
2 - Table of contents
3 - List of figures and tables
4 - Introduction
5 - Summary
6 - Conclusions
7 - References
8 - Appendixes

INORGANIC DATA VALIDATION CHECKLIST

Laboratory _____

SDG Number _____

This data package has been reviewed and the Quality Assurance and performance data summarized. The review is based on the following information; Holding times, calibration, blanks, spikes, duplicates, and sample result verification.

Any exceedance in the QC limits are documented in the attached summary sheets. The following qualifiers may be applied:

J = Positive result at an estimated value

R = Data is unreliable due to significant QC problems

B = Blank contamination

Data Reviewer _____

INORGANIC ANALYSES

1. Holding Times/Preservation Requirements

Holding times will be evaluated.

2. Calibration

Was the instrument calibrated daily or at each time it was set up?

Yes No

JCP Analyses - Were a blank and one standard used in establishing the analytical curve?

Yes No

Atomic absorption Analysis - Were three standards, one of which must be at the method detection limit, and a blank used in establishing an analytical curve with correlation coefficient of >0.995 ?

Yes No

Mercury Analyses - Were four standards and a blank used in establishing the analytical curve with a correlation coefficient of > 0.995 ?

Yes No

Cyanide Analyses - Was a blank and three standards used in establishing the analytical curve with a correlation coefficient of >0.995 ?

Yes No

Cyanide Analyses - Was a midrange standard distilled and analyzed?

Yes No

Action: If the minimum number of standards have not been used, or if the instrument was not calibrated daily or each time it was set up, reject all associated data (R).

Action: If the midrange standard for cyanide was not distilled qualify all positive values

estimated

(J).

Action: If one of the standards for AA was not ran at the IDL note in the comments section and use professional judgment in qualifying the samples.

Action: If the correlation coefficient less than 0.995 flag all detects with a (J) as estimated.

3. Initial and Continuing Calibration Verification

Are initial and continuing calibration forms present and complete for every metal and cyanide?

Yes No

Action: If no, please request this information from the laboratory

Are all calibration standards (initial and continuing) within control limits?

Metals	90 - 110%	<input type="checkbox"/> Yes	<input type="checkbox"/> No
Mercury	80 - 120%	<input type="checkbox"/> Yes	<input type="checkbox"/> No
Cyanide	85 - 115%	<input type="checkbox"/> Yes	<input type="checkbox"/> No

Action: If ICV or CCV is between the following ranges qualify all detected analyses as estimated, "J"

Metals	75% - 89%, 111% - 125%
Mercury	65% - 79%, 121% - 135%
Cyanide	70% - 84%, 116% - 130%

If ICV or CCV is below the following ranges qualify both detected and non-detected samples as rejected, "R".

Metals	75%
Mercury	65%
Cyanide	70%

If ICV or CCV is above the following ranges qualify all detected results rejected, "R".

Metals	125%
Mercury	130%
Cyanide	135%

Note: CCV applies to all sample analyses ran before and after to the next good CCV.

4. Blanks

Was an initial and continuing blank analyzed? Yes No

Was a prep blank analyzed for each batch and each matrix type?
 Yes No

Were contaminants found in the blanks? Yes No

Action: If samples associated ~ with the blank have an analyze concentration greater than the IDL but less than five times the blank concentration, qualify the data as (B).

The actual comparison of blank and sample results--will--e--ased~ on-actual instrument value, particularly for soil matrix. This approach will eliminate variability in results due to sample weight/volume, percent moisture, etc. which vary from sample to sample. In instances where more than one blank is associated with a given sample, qualification should be based upon associated blank having the highest concentration of a contaminant.

Note: Field blanks will be evaluated and applied as a separate step in the validation process.

5. ICP Interference Check Sample

Was ICS analyzed at beginning and end of run or at least twice every 8 hours?
 Yes No

Action: If no, flag all detects as estimated (J) all samples for which Al, Ca, Fe, or Mg is higher than in ICP.

Note: Not required for furnace AA, flame AA, mercury, cyanide and Ca, Mg, K and Na.

Are all interference check sample results inside of control limits (+/-20%)?
 Yes No

If no, is concentration of Al, Ca, Fe, or Mg in the sample lower than the respective concentration in ICP? Yes No

Action: If no, flag as estimated (J) those positive results for which ICP recovery is greater than 121 %; flag all sample results as estimated (J) if ICP recovery falls within 50-79%; Flag all sample results as unreliable (R) for which the ICP recovery is less than 50%.

Note: If results greater than the IDL are observed for elements which are not present in the EPA provided ICP solution, the possibility of false positive exists. An evaluation of the associated sample data for the affected elements should be made.

Action: For samples with comparable or higher levels of interference and with analyze concentrations that approximate those levels found in the ICP (false positive), qualify sample results >IDL and less than the values observed as estimate (J).

6. Laboratory Control Sample (LCS)

Are LCS values within Control Limits? Yes No

Action: For aqueous LCS, if the LCS recovery for any analyze falls within the range of 50-79% or > 120%, qualify results >IDL as estimated (J). If the LCS recovery results are <50%, qualify both detects and non-detects the data for these samples as unreliable (R).

Action: For solid LCS values above or below the control limits, qualify all detected values estimated "J".

Note: All aqueous LCS results must fall within the control limits of 80-120% except Antimony and Silver which have no control limits. All solid LCS results must fall within control limits as established by EPA.

Was one LCS prepared and analyzed at the beginning for:

Water Samples?	<input type="checkbox"/> Yes	<input type="checkbox"/> No
Solid Samples?	<input type="checkbox"/> Yes	<input type="checkbox"/> No

Action: If no for any of the above, note in the comments section and qualify all detects estimated "J" for which no LCS was analyzed.

7. Duplicate Sample Analysis

Present for each 20 samples? Yes No
 Present for each Matrix type? Yes No

Action: If no for any of the above, flag as estimated (J) all data greater than IDL for which duplicate sample was not analyzed.

Note:

. If one duplicate sample was analyzed for more than 20 samples, then the first 20 samples do not have to be flagged as estimated.

2. If percent solids for soil sample and its duplicate differ by more than 1%, calculate %RPD based on wet weight.

Are all values within control limits?

;for sample values greater than 5x CRDL? Yes No
 ;for sample values less than 5x CRDL? Yes No

Note: For samples values greater than 5x CRDL must have %RPD of less than 20% for aqueous samples and %RPD of less than 35% for soil samples. For sample values less than 5x CRDL must be within 2x the CRDL for aqueous samples and within 5x the CRDL for soils.

Action: If a duplicate analysis results for an analyze fall outside the control limits, qualify all positive results for that analyze in all associated samples of the same matrix estimated "J".

8. Matrix Spike Analysis

Are all values within control limits 75-125%? Yes No

Note: Spike recovery limits do not apply when sample concentration exceeds in the QC sample the spike concentration by a factor of 4 or more.

Action: If the spike recovery is > 125 % or < 75 % and the sample results are greater than IDL qualify the data for these samples are as estimated, "J". If spike recovery is less than 30% reject "R" all non-detects.

9. Furnace Atomic Absorption (AA)

Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?
 Yes No

Action: If no, use professional judgment to qualify data.

Are the Spike recoveries within control limits 85% and 115%?
 Yes No

Do the duplicate injection readings have Relative Standard Deviation (RSD) or Coefficient of Variation (CV) of 20% or less for sample concentrations above the CRDL?
 Yes No

If the RSD or CV were above 20%, was the sample reanalyzed?
 Yes No N/A

Action: If the sample was not reanalyzed, or if upon reanalysis the criteria are not met, qualify the associated data estimated (J).

If the post digestion spike recovery is between 10%-85% or above 115%, qualify result >IDL as estimated (J).

If post digestion spike recovery is <10%, qualify results <IDL unusable (R) and detects as estimated "J".

Are any correlation Coefficients for MSA below 0.995?

Yes No

Action: If MSA correlation is less than 0.995 qualify detects for associated analyses estimated "J"

Was a MSA required for any analyze due to post digestion spike recovery, but not performed?

Yes No

Action: Qualify all detects for associated sample analyses estimated "J".

10. Serial Dilution

Do sample results for the serial dilution agree within 20% of the original results?

Yes No

Action: When criteria are not Met, qualify the associated data as estimated (J), (only for sample values above 50 times the IDL).

Note: If the analyze concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a 5-fold dilution must agree within 10% Difference (%D) or the original results.

APPENDIX F
SPECIAL PARAMETERS

1. Chain-of-Custody Records and Request for Analysis (C-O-C/RFA)

Are C-O-C/Request For Analysis Records present for all samples?

 Yes No N/A

Do the C-O-C/Request For Analysis or Lab Narrative indicate any problems with sample receipt condition of samples, analytical problems, or special circumstances affecting the quality of the data?

 Yes No N/A

Action: Use professional judgment to evaluate the effect on the quality of the data

Note: Holding times will be reviewed using the IRDMIS system.

2. Matrix Spikes

Was a Matrix Spike or-Matrix Spike/Matrix Spike Duplicate ran?

 Yes No N/A

Action: If any matrix spike data are missing, note in case narrative.

Were Matrix Spike Recoveries between 75-125%? Yes No N/A

ACTION: If recoveries are outside QC limits qualify all associated detected sample results estimated "J". Professional judgment may be used to reject sample results if spike recoveries are grossly outside QC limits.

3. Duplicates

Was a Duplicate ran?

 Yes No N/A

ACTION: If no note in case narrative.

Were duplicate results within QC limits (Soil 35%,2 * CRDL, Water 20%,1 ~ CRDL)?

Yes No N/A

ACTION: If recoveries are outside QC criteria qualify all associated detected results estimated "J".

4. Blanks

Has a method blank been analyzed at least once per analytical batch or per 20 samples?

Yes No N/A

Action: If any method blank data are missing, note in the case narrative.

Do any method blanks have positive results?

Yes No N/A

Action: For sample analyses sample concentrations less than five times the blank value qualify "B" in both the USAT and EPA qualification fields. Use the largest value from all the associated blanks

Note: Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems. Field blank }c contamination is not to be applied at this time. This will be done as a separate step in the validation process. Do not apply qualifiers to rinse blanks, (which are denoted as RNSW under Site Type on the COC Form).

5. Laboratory Control Sample (LCS)

Is the Laboratory Control Sample (LCS) within quality control limits?

Yes No N/A

Action: If the LCS is outside control limits, qualify all detects as estimated "J".

APPENDIX E

**QUANTERRA REFERENCE DATA
SUMMARY
(METHOD DETECTION LIMITS AND
METHOD RECOVERY LIMITS)**

QUANTERRA REFERENCE DATA SUMMARY

Target Analyte List: Q: CLP MSSemi TCL Standard List

Matrix: SOLID
 Extraction: SOXHLET (NONE,Na2SO4)
 Method: Base/Neutrals and Acids (8270C)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2101		Detection Limits			Run Date	Check List 1660					Spike List 1661				
Compound	RL	Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	330	ug/kg	18	ug/kg	19980202	1700	ug/kg	53	101	24	1700	ug/kg	47	145	28
Acenaphthylene	330	ug/kg	16	ug/kg	19980123										
Anthracene	330	ug/kg	22	ug/kg	19980202										
Benzo(a)anthracene	330	ug/kg	34	ug/kg	19980306										
Benzo(b)fluoranthene	330	ug/kg	36	ug/kg	19980306										
Benzo(k)fluoranthene	330	ug/kg	40	ug/kg	19980306										
Benzo(ghi)perylene	330	ug/kg	48	ug/kg	19980306										
Benzo(a)pyrene	330	ug/kg	41	ug/kg	19980306										
bis(2-Chloroethoxy)methane	330	ug/kg	21	ug/kg	19980202										
bis(2-Chloroethyl) ether	330	ug/kg	33	ug/kg	19980128										
bis(2-Ethylhexyl) phthalate	330	ug/kg	37	ug/kg	19980122										
4-Bromophenyl phenyl ether	330	ug/kg	23	ug/kg	19980202										
Butyl benzyl phthalate	330	ug/kg	34	ug/kg	19980306										
Carbazole	330	ug/kg	24	ug/kg	19980122										
4-Chloroaniline	330	ug/kg	39	ug/kg	19980202										
4-Chloro-3-methylphenol	330	ug/kg	26	ug/kg	19980202	2500	ug/kg	46	106	30	2500	ug/kg	22	147	37
2-Chloronaphthalene	330	ug/kg	13	ug/kg	19980202										
2-Chlorophenol	330	ug/kg	29	ug/kg	19980128	2500	ug/kg	44	103	30	2500	ug/kg	23	134	29
4-Chlorophenyl phenyl ether	330	ug/kg	17	ug/kg	19980202										
Chrysene	330	ug/kg	29	ug/kg	19980202										
Dibenz(a,h)anthracene	330	ug/kg	52	ug/kg	19980306										
Dibenzofuran	330	ug/kg	25	ug/kg	19980202										
Di-n-butyl phthalate	330	ug/kg	19	ug/kg	19980202										
1,2-Dichlorobenzene	330	ug/kg	24	ug/kg	19980128										
1,3-Dichlorobenzene	330	ug/kg	28	ug/kg	19980128										
1,4-Dichlorobenzene	330	ug/kg	18	ug/kg	19980128	1700	ug/kg	51	95	22	1700	ug/kg	20	124	32
3,3'-Dichlorobenzidine	1600	ug/kg	29	ug/kg	19970303										
2,4-Dichlorophenol	330	ug/kg	26	ug/kg	19980202										
Diethyl phthalate	330	ug/kg	20	ug/kg	19980128										
2,4-Dimethylphenol	330	ug/kg	73	ug/kg	19980202										
Dimethyl phthalate	330	ug/kg	16	ug/kg	19980202										
4,6-Dinitro-2-methylphenol	1600	ug/kg	26	ug/kg	19980202										
2,4-Dinitrophenol	1600	ug/kg	71	ug/kg	19980311										
2,4-Dinitrotoluene	330	ug/kg	31	ug/kg	19980202	1700	ug/kg	54	129	37	1700	ug/kg	39	139	22
2,6-Dinitrotoluene	330	ug/kg	32	ug/kg	19980306										
Di-n-octyl phthalate	330	ug/kg	47	ug/kg	19980306										

Target Analyte List: Q: CLP MSSemi TCL Standard List

Matrix: SOLID
 Extraction: SOXHLET (NONE,Na2SO4)
 Method: Base/Neutrals and Acids (8270C)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2101		Detection Limits			Run Date	Check List 1660					Spike List 1661				
Compound	RL	Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Fluoranthene	330	ug/kg	26	ug/kg	19980128										
Fluorene	330	ug/kg	20	ug/kg	19980202										
Hexachlorobenzene	330	ug/kg	34	ug/kg	19980202										
Hexachlorobutadiene	330	ug/kg	40	ug/kg	19980202										
Hexachlorocyclopentadiene	1600	ug/kg	26	ug/kg	19980122										
Hexachloroethane	330	ug/kg	22	ug/kg	19980128										
Indeno(1,2,3-cd)pyrene	330	ug/kg	41	ug/kg	19980306										
Isophorone	330	ug/kg	14	ug/kg	19980202										
2-Methylnaphthalene	330	ug/kg	16	ug/kg	19980128										
2-Methylphenol	330	ug/kg	31	ug/kg	19980128										
4-Methylphenol	330	ug/kg	31	ug/kg	19980202										
Naphthalene	330	ug/kg	14	ug/kg	19980202										
2-Nitroaniline	1600	ug/kg	26	ug/kg	19980202										
3-Nitroaniline	1600	ug/kg	31	ug/kg	19980122										
4-Nitroaniline	1600	ug/kg	53	ug/kg	19980122										
Nitrobenzene	330	ug/kg	40	ug/kg	19980202										
2-Nitrophenol	330	ug/kg	26	ug/kg	19980202										
4-Nitrophenol	1600	ug/kg	120	ug/kg	19980311	2500	ug/kg	16	192	88	2500	ug/kg	10	132	47
N-Nitrosodiphenylamine	330	ug/kg	28	ug/kg	19980128										
N-Nitrosodi-n-propylamine	330	ug/kg	29	ug/kg	19980202	1700	ug/kg	39	95	28	1700	ug/kg	10	230	55
Pentachlorophenol	330	ug/kg	27	ug/kg	19980122	2500	ug/kg	24	115	45	2500	ug/kg	14	176	49
Phenanthrene	330	ug/kg	19	ug/kg	19980202										
Phenol	330	ug/kg	25	ug/kg	19980202	2500	ug/kg	36	103	34	2500	ug/kg	10	112	23
2,2'-Oxybis(1-Chloropropane)	330	ug/kg	18	ug/kg	19980128										
Pyrene	330	ug/kg	32	ug/kg	19980202	1700	ug/kg	46	147	51	1700	ug/kg	52	115	25
1,2,4-Trichlorobenzene	330	ug/kg	17	ug/kg	19980128	1700	ug/kg	51	101	25	1700	ug/kg	44	142	28
2,4,5-Trichlorophenol	330	ug/kg	25	ug/kg	19980202										
2,4,6-Trichlorophenol	330	ug/kg	18	ug/kg	19980202										
2-Fluorobiphenyl						1660	ug/kg	30	115	0	1660	ug/kg	30	115	0
2-Fluorophenol						2500	ug/kg	25	121	0	2500	ug/kg	25	121	0
2,4,6-Tribromophenol						2500	ug/kg	19	122	0	2500	ug/kg	19	122	0
Nitrobenzene-d5						1660	ug/kg	23	120	0	1660	ug/kg	23	120	0
Phenol-d5						1500	ug/kg	24	113	0	2500	ug/kg	24	113	0
Terphenyl-d14						1660	ug/kg	18	137	0	1660	ug/kg	18	137	0

Quanterra Reference Data Summary

Target Analyte List: NC: TCL Pesticide 8081A List	Matrix:	SOLID
	Extraction:	SOXHLET (NONE,Na2SO4)
	Method:	Pesticides (8081A)
	QC Program:	STANDARD TEST SET
	Location:	Quanterra - North Canton

Target List 4115		Detection Limits				Check List 912					Spike List 913				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aldrin	1.7	ug/kg	0.37	ug/kg	19980311	6.67	ug/kg	43	116	37	6.67	ug/kg	31	123	42
alpha-BHC	1.7	ug/kg	0.17	ug/kg	19980311										
beta-BHC	1.7	ug/kg	0.38	ug/kg	19980311										
delta-BHC	1.7	ug/kg	0.28	ug/kg	19980311										
gamma-BHC (Lindane)	1.7	ug/kg	0.21	ug/kg	19980311										
alpha-Chlordane	1.7	ug/kg	0.68	ug/kg	19980311										
gamma-Chlordane	1.7	ug/kg	0.32	ug/kg	19980311										
4,4'-DDD	1.7	ug/kg	0.4	ug/kg	19980303										
4,4'-DDE	1.7	ug/kg	0.55	ug/kg	19980311										
4,4'-DDT	1.7	ug/kg	0.86	ug/kg	19980311	16.67	ug/kg	52	128	38	16.67	ug/kg	10	151	50
Dieldrin	1.7	ug/kg	0.5	ug/kg	19980303	16.67	ug/kg	62	107	23	16.67	ug/kg	32	145	43
Endosulfan I	1.7	ug/kg	0.46	ug/kg	19980311										
Endosulfan II	1.7	ug/kg	0.47	ug/kg	19980311										
Endosulfan sulfate	1.7	ug/kg	0.49	ug/kg	19980311										
Endrin	1.7	ug/kg	0.43	ug/kg	19980303	16.67	ug/kg	64	127	31	16.67	ug/kg	32	137	45
Endrin aldehyde	1.7	ug/kg	0.92	ug/kg	19980311										
Endrin ketone	1.7	ug/kg	0.65	ug/kg	19980304										
Heptachlor	1.7	ug/kg	0.23	ug/kg	19980303	6.67	ug/kg	53	130	38	6.67	ug/kg	24	168	73
Heptachlor epoxide	1.7	ug/kg	0.3	ug/kg	19980304										
Methoxychlor	3.3	ug/kg	2.8	ug/kg	19980303										
Toxaphene	67	ug/kg	10	ug/kg	19980416										
Decachlorobiphenyl						6.67	ug/kg	10	138	0	6.67	ug/kg	10	138	0
Tetrachloro-m-xylene						6.67	ug/kg	10	129	0	6.67	ug/kg	10	129	0

Quanterra Report - GC Data Summary

Target Analyte List: Q: CLP MSVOA TCL Standard List

Matrix: SOLID
 Extraction: PURGE AND TRAP - 5 mL purge
 Method: Volatile Organics, GC/MS (8260B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2001		Detection Limits			Run Date	Check List 902					Spike List 903				
Compound	RL	Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acetone	20	ug/kg	2.3	ug/kg	19980324										
Benzene	5	ug/kg	0.25	ug/kg	19980130	20	ug/kg	85	120	13	50	ug/kg	84	121	13
Bromodichloromethane	5	ug/kg	0.21	ug/kg	19980130										
Bromoform	5	ug/kg	0.27	ug/kg	19980130										
Bromomethane	10	ug/kg	0.9	ug/kg	19980130										
2-Butanone	20	ug/kg	1	ug/kg	19980130										
Carbon disulfide	5	ug/kg	0.35	ug/kg	19980130										
Carbon tetrachloride	5	ug/kg	0.11	ug/kg	19980130										
Chlorobenzene	5	ug/kg	0.22	ug/kg	19980130	20	ug/kg	88	119	22	50	ug/kg	76	122	22
Dibromochloromethane	5	ug/kg	0.21	ug/kg	19980130										
Chloroethane	10	ug/kg	0.45	ug/kg	19980130										
Chloroform	5	ug/kg	0.25	ug/kg	19980130										
Chloromethane	10	ug/kg	0.56	ug/kg	19980130										
1,1-Dichloroethane	5	ug/kg	0.2	ug/kg	19980130										
1,2-Dichloroethane	5	ug/kg	0.24	ug/kg	19980130										
1,1-Dichloroethene	5	ug/kg	0.2	ug/kg	19980130	20	ug/kg	76	128	17	50	ug/kg	75	131	17
1,2-Dichloroethene (total)	5	ug/kg	1.1	ug/kg	19980130										
1,2-Dichloropropane	5	ug/kg	0.29	ug/kg	19980130										
cis-1,3-Dichloropropene	5	ug/kg	0.25	ug/kg	19980130										
trans-1,3-Dichloropropene	5	ug/kg	0.11	ug/kg	19980130										
Ethylbenzene	5	ug/kg	0.27	ug/kg	19980130										
2-Hexanone	20	ug/kg	0.7	ug/kg	19980130										
Methylene chloride	5	ug/kg	0.28	ug/kg	19980130										
4-Methyl-2-pentanone	20	ug/kg	0.46	ug/kg	19980130										
Styrene	5	ug/kg	0.24	ug/kg	19980130										
1,1,2,2-Tetrachloroethane	5	ug/kg	0.35	ug/kg	19980130										
Tetrachloroethene	5	ug/kg	0.2	ug/kg	19980130										
Toluene	5	ug/kg	0.25	ug/kg	19980130	20	ug/kg	86	118	23	50	ug/kg	79	129	23
1,1,1-Trichloroethane	5	ug/kg	0.12	ug/kg	19980130										
1,1,2-Trichloroethane	5	ug/kg	0.2	ug/kg	19980130										
Trichloroethene	5	ug/kg	0.23	ug/kg	19980130	20	ug/kg	86	116	17	50	ug/kg	78	121	17
Vinyl chloride	10	ug/kg	0.15	ug/kg	19980130										
Xylenes (total)	5	ug/kg	0.72	ug/kg	19980130										
Bromofluorobenzene						50	ug/kg	60	137	0	50	ug/kg	60	137	0
1,2-Dichloroethane-d4						50	ug/kg	75	117	0	50	ug/kg	75	117	0
Toluene-d8						50	ug/kg	86	122	0	50	ug/kg	86	122	0

Target Analyte List: Q: CLP MSVOA TCL Standard List

Matrix: SOLID
Extraction: PURGE AND TRAP - 5 mL purge
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: Quanterra - North Canton

Target List 2001		Detection Limits				Check List 902				Spike List 903					
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Dibromofluoromethane						50	ug/kg	70	135	0	50	ug/kg	70	135	0

QUANTERRA REGIST CG Data Summary

Target Analyte List: Q: CLP ICP Metals TAL Standard List

Matrix: SOLID
 Extraction: METALS, TOTAL - Soils
 Method: Inductively Coupled Plasma (6010B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Compound	Target List 2600		Detection Limits		Run Date	Check List 906					Spike List 907				
	RL	Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	20	mg/kg	4.4	mg/kg	19980209	200	mg/kg	80	113	20	200	mg/kg	80	120	20
Antimony	6	mg/kg	2.1	mg/kg	19980209	50	mg/kg	80	104	15	50	mg/kg	80	120	20
Barium	20	mg/kg	0.22	mg/kg	19980209	200	mg/kg	80	109	15	200	mg/kg	80	120	20
Beryllium	0.5	mg/kg	0.067	mg/kg	19980209	5	mg/kg	80	105	14	5	mg/kg	80	120	20
Cadmium	0.5	mg/kg	0.49	mg/kg	19980209	5	mg/kg	80	112	16	5	mg/kg	80	120	20
Calcium	500	mg/kg	14	mg/kg	19980206	5000	mg/kg	80	109	16	5000	mg/kg	80	120	20
Chromium	1	mg/kg	0.54	mg/kg	19980209	20	mg/kg	81	116	17	20	mg/kg	80	120	20
Cobalt	5	mg/kg	0.59	mg/kg	19980209	50	mg/kg	80	104	14	50	mg/kg	80	120	20
Copper	2.5	mg/kg	0.31	mg/kg	19980209	25	mg/kg	80	113	18	25	mg/kg	80	120	20
Iron	10	mg/kg	4.9	mg/kg	19980206	100	mg/kg	80	120	20	100	mg/kg	80	120	20
Magnesium	500	mg/kg	11	mg/kg	19980209	5000	mg/kg	80	109	17	5000	mg/kg	80	120	20
Manganese	1.5	mg/kg	0.082	mg/kg	19980224	50	mg/kg	80	114	17	50	mg/kg	80	120	20
Nickel	4	mg/kg	1.1	mg/kg	19980209	50	mg/kg	80	112	16	50	mg/kg	80	120	20
Potassium	500	mg/kg	8	mg/kg	19980209	5000	mg/kg	80	103	12	5000	mg/kg	80	120	20
Silver	1	mg/kg	0.42	mg/kg	19980209	5	mg/kg	81	120	20	5	mg/kg	80	120	20
Sodium	500	mg/kg	14	mg/kg	19980209	5000	mg/kg	80	107	14	5000	mg/kg	80	120	20
Vanadium	5	mg/kg	0.57	mg/kg	19980209	50	mg/kg	80	111	17	50	mg/kg	80	120	20
Zinc	2	mg/kg	1.2	mg/kg	19980209	50	mg/kg	80	120	21	50	mg/kg	80	120	20

QUANTERRA REFERENCE DATA SUMMARY

Target Analyte List: All Analytes	Matrix: SOLID Extraction: METALS, TOTAL - Soils Method: Inductively Coupled Plasma (6010B Trace) QC Program: STANDARD TEST SET Location: Quanterra - North Canton
-----------------------------------	---

Analyte List		Detection Limits			Run Date	Check List 904					Spike List 905				
Compound	RL	Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Antimony	1.0	mg/kg	0.24	mg/kg	19980224	50	mg/kg	80	120	20	50	mg/kg	80	120	20
Arsenic	1.0	mg/kg	0.24	mg/kg	19980206	200	mg/kg	80	120	20	200	mg/kg	80	120	20
Cadmium	0.2	mg/kg	0.045	mg/kg	19980224	5	mg/kg	80	120	20	5	mg/kg	80	120	20
Chromium	0.5	mg/kg	0.3	mg/kg	19980224	20	mg/kg	80	120	20	20	mg/kg	80	120	20
Cobalt	5	mg/kg	0.15	mg/kg	19980224	50	mg/kg	80	120	20	50	mg/kg	80	120	20
Lead	0.3	mg/kg	0.19	mg/kg	19980325	50	mg/kg	80	120	20	50	mg/kg	80	120	20
Molybdenum	1.0	mg/kg	0.31	mg/kg	19980224	100	mg/kg	80	120	20	100	mg/kg	80	120	20
Selenium	0.5	mg/kg	0.49	mg/kg	19980325	200	mg/kg	80	120	20	200	mg/kg	80	120	20
Silver	0.5	mg/kg	0.18	mg/kg	19980325	5	mg/kg	80	120	20	5	mg/kg	80	120	20
Thallium	1.0	mg/kg	0.65	mg/kg	19980224	200	mg/kg	80	120	20	200	mg/kg	80	120	20
Vanadium	5	mg/kg	0.12	mg/kg	19980206	50	mg/kg	80	120	20	50	mg/kg	80	120	20

QUANTERRA REGRE Data Summary

Target Analyte List: All Analytes	Matrix: SOLID Extraction: METALS, TOTAL (Method Exclusive) - Solids Method: Mercury (7471A, Cold Vapor) - Solids QC Program: STANDARD TEST SET Location: Quanterra - North Canton
-----------------------------------	---

Analyte List		Detection Limits			Check List 1021					Spike List 1026					
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Mercury	0.1	mg/kg	0.0047	mg/kg	19990120	.833333	ng/kg	70	130	20	0.1666	mg/kg	70	130	20

Quanterra Reference Data Summary

Target Analyte List: KNX: HPLC 8330 STANDARD LIST

Matrix: SOLID
 Extraction: SONICATION - Low Level
 Method: Nitroaromatics & Nitramines "Explosives" (8330)
 QC Program: STANDARD TEST SET
 Location: Quanterra - Knoxville

Target List 4832		Detection Limits			Run Date	Check List 8215					Spike List 8216				
Compound	RL	Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
4-Amino-2,6-dinitrotoluene	0.25	mg/kg	0.02	mg/kg	19980217	3.0	mg/kg	81	123	15	3.0	mg/kg	71	123	15
2-Amino-4,6-dinitrotoluene	0.25	mg/kg	0.03	mg/kg	19980217	3.0	mg/kg	80	121	16	3.0	mg/kg	75	121	16
1,3-Dinitrobenzene	0.25	mg/kg	0.02	mg/kg	19980217	3.0	mg/kg	82	117	15	3.0	mg/kg	82	117	15
2,4-Dinitrotoluene	0.25	mg/kg	0.03	mg/kg	19980217	3.0	mg/kg	81	120	15	3.0	mg/kg	81	120	15
2,6-Dinitrotoluene	0.25	mg/kg	0.06	mg/kg	19980217	3.0	mg/kg	82	121	15	3.0	mg/kg	78	125	15
HMX	0.50	mg/kg	0.05	mg/kg	19980217	3.0	mg/kg	77	122	17	3.0	mg/kg	72	122	17
Nitrobenzene	0.25	mg/kg	0.10	mg/kg	19980217	3.0	mg/kg	83	117	15	3.0	mg/kg	83	117	15
2-Nitrotoluene	0.25	mg/kg	0.02	mg/kg	19980217	3.0	mg/kg	81	117	15	3.0	mg/kg	81	117	15
4-Nitrotoluene	0.25	mg/kg	0.02	mg/kg	19980217	3.0	mg/kg	81	117	15	3.0	mg/kg	80	117	15
3-Nitrotoluene	0.25	mg/kg	0.03	mg/kg	19980217	3.0	mg/kg	80	118	15	3.0	mg/kg	80	118	15
RDX	0.50	mg/kg	0.02	mg/kg	19980217	3.0	mg/kg	83	121	16	3.0	mg/kg	71	121	16
Tetryl	0.65	mg/kg	0.03	mg/kg	19980217	3.0	mg/kg	51	120	20	3.0	mg/kg	51	124	20
1,3,5-Trinitrobenzene	0.25	mg/kg	0.02	mg/kg	19980217	3.0	mg/kg	81	120	15	3.0	mg/kg	81	120	15
2,4,6-Trinitrotoluene	0.25	mg/kg	0.02	mg/kg	19980217	3.0	mg/kg	74	126	15	3.0	mg/kg	74	126	15
1-Chloro-3-nitrobenzene						1.77	mg/kg	76	124	0	1.77	mg/kg	76	124	0

Quanterra Reference Data Summary

Target Analyte List: Q: SW846 Method 8021 Standard List

Matrix: SOLID
 Extraction: PURGE AND TRAP - 5 mL purge
 Method: Volatile Organics (8021B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2313		Detection Limits				Check List 914				Spike List 915					
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Benzene	1	ug/kg	0.27	ug/kg	19980303			69	132	20			62	150	33
Bromobenzene	1	ug/kg	0.45	ug/kg	19980303										
Bromochloromethane	1	ug/kg	0.17	ug/kg	19980303										
Bromodichloromethane	1	ug/kg	0.23	ug/kg	19980303										
Bromoform	1	ug/kg	0.35	ug/kg	19980303										
Bromomethane	1	ug/kg	0.16	ug/kg	19980303										
n-Butylbenzene	1	ug/kg	0.45	ug/kg	19980303										
sec-Butylbenzene	1	ug/kg	0.47	ug/kg	19980303										
tert-Butylbenzene	1	ug/kg	0.47	ug/kg	19980303										
Carbon tetrachloride	1	ug/kg	0.4	ug/kg	19980303										
Chlorobenzene	1	ug/kg	0.65	ug/kg	19970609			51	131	20			10	142	32
Chlorodibromomethane	1	ug/kg	0.32	ug/kg	19980303										
Chloroethane	1	ug/kg	0.15	ug/kg	19980303										
Chloroform	1	ug/kg	0.26	ug/kg	19980303										
Chloromethane	1	ug/kg	0.15	ug/kg	19980303										
2-Chlorotoluene	1	ug/kg	0.41	ug/kg	19980303										
4-Chlorotoluene	1	ug/kg	0.52	ug/kg	19980303										
1,2-Dibromo-3-chloropropane (DBCP)	1	ug/kg			0										
1,2-Dibromoethane (EDB)	1	ug/kg	0.24	ug/kg	19980303										
Dibromomethane	1	ug/kg	0.24	ug/kg	19980303										
1,2-Dichlorobenzene	1	ug/kg	0.69	ug/kg	19970609										
1,3-Dichlorobenzene	1	ug/kg	0.78	ug/kg	19970609										
1,4-Dichlorobenzene	1	ug/kg	0.76	ug/kg	19970609										
Dichlorodifluoromethane	1	ug/kg	0.18	ug/kg	19980303										
1,1-Dichloroethane	1	ug/kg	0.18	ug/kg	19980303										
1,2-Dichloroethane	1	ug/kg	0.21	ug/kg	19980303										
cis-1,2-Dichloroethene	1	ug/kg	0.67	ug/kg	19980303										
trans-1,2-Dichloroethene	1	ug/kg	0.21	ug/kg	19980303										
1,1-Dichloroethene	1	ug/kg	0.82	ug/kg	19970609			29	140	20			10	203	38
1,2-Dichloropropane	1	ug/kg	0.2	ug/kg	19980303										
1,3-Dichloropropane	1	ug/kg	0.17	ug/kg	19980303										
2,2-Dichloropropane	1	ug/kg	0.45	ug/kg	19980303										
cis-1,3-Dichloropropene	1	ug/kg	0.55	ug/kg	19980303										
trans-1,3-Dichloropropene	1	ug/kg	0.54	ug/kg	19980303										
1,1-Dichloropropene	1	ug/kg	0.4	ug/kg	19980303										
Ethylbenzene	1	ug/kg	0.46	ug/kg	19980303										

Target Analyte List: Q: SW846 Method 8021 Standard List

Matrix: SOLID
 Extraction: PURGE AND TRAP - 5 mL purge
 Method: Volatile Organics (8021B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2313		Detection Limits				Check List 914					Spike List 915				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Hexachlorobutadiene	1	ug/kg	0.48	ug/kg	19980303										
Isopropylbenzene	1	ug/kg	0.56	ug/kg	19980303										
p-Isopropyltoluene	1	ug/kg	0.96	ug/kg	19980303										
Methylene chloride	5	ug/kg	0.15	ug/kg	19980303										
Naphthalene	1	ug/kg	0.25	ug/kg	19980303										
n-Propylbenzene	1	ug/kg	0.43	ug/kg	19980303										
Styrene	1	ug/kg			0										
1,1,1,2-Tetrachloroethane	1	ug/kg	0.18	ug/kg	19980303										
1,1,2,2-Tetrachloroethane	1	ug/kg	0.88	ug/kg	19980303										
Tetrachloroethene	1	ug/kg			0										
Toluene	1	ug/kg	0.41	ug/kg	19980303			66	129	20			50	142	40
1,2,3-Trichlorobenzene	1	ug/kg	0.88	ug/kg	19980303										
1,2,4-Trichlorobenzene	1	ug/kg	0.44	ug/kg	19980303										
1,1,1-Trichloroethane	1	ug/kg	0.22	ug/kg	19980303										
1,1,2-Trichloroethane	1	ug/kg	0.31	ug/kg	19980303										
Trichloroethene	1	ug/kg	0.64	ug/kg	19970609			10	216	20			10	216	45
Trichlorofluoromethane	1	ug/kg	0.17	ug/kg	19980303										
1,2,3-Trichloropropane	1	ug/kg			0										
1,2,4-Trimethylbenzene	1	ug/kg	0.47	ug/kg	19980303										
1,3,5-Trimethylbenzene	1	ug/kg			0										
Vinyl chloride	1	ug/kg	0.14	ug/kg	19980303										
Xylenes (total)	1	ug/kg			0										
1,4-Dichlorobutane								50	150	0			50	150	0
Trifluorotoluene								50	150	0			50	150	0

QUANTERRA REPORT - Data Summary

Target Analyte List: Q: Aroclors Only Standard List

Matrix: SOLID
 Extraction: SOXHLET (Na2SO4) w/ACID STRIP (PCB)
 Method: PCBs (8082)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2207		Detection Limits				Check List 1832					Spike List 1833				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aroclor 1016	33	ug/kg	12	ug/kg	19980315	333	ug/kg	60	133	37	333	ug/kg	44	139	28
Aroclor 1221	33	ug/kg	29	ug/kg	19980507										
Aroclor 1232	33	ug/kg	8	ug/kg	19980315										
Aroclor 1242	33	ug/kg	18	ug/kg	19980315										
Aroclor 1248	33	ug/kg	3.9	ug/kg	19980319										
Aroclor 1254	33	ug/kg	7.3	ug/kg	19980507										
Aroclor 1260	33	ug/kg	7.3	ug/kg	19980507	333	ug/kg	59	129	35	333	ug/kg	44	139	28
Decachlorobiphenyl						6.67	ug/kg	10	138	0	6.67	ug/kg	10	138	0
Tetrachloro-m-xylene						6.67	ug/kg	10	129	0	6.67	ug/kg	10	129	0

QUANTERRA REFERENCE DATA SUMMARY

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: PURGE AND TRAP - 5 mL purge
 Method: Hydrocarbons, Volatile Petroleum (8015B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Analyte List		Detection Limits				Check List 1600					Spike List 1601				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
TPH (as Gasoline)	100	ug/kg			0			38	120	49			10	114	49

Quanterra Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: SONICATION - Special Low Level (>sample mass or <final
 Method: Hydrocarbons, Extractable Petroleum (8015B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Analyte List Compound	RL	Detection Limits			Run Date	Check List 1801			Spike List 1802					
		Units	MDL	Units		Amt	Units	LCL UCL RPD	Amt	Units	LCL UCL RPD			
TPH (Extractables)	3	mg/kg			0									
Total Petroleum Hydrocarbons - Extrac	3	mg/kg			0			38	120	49		10	114	49
Total Petroleum Hydrocarbons	3	mg/kg			0									
TPH (as Diesel)	3	mg/kg			0									
TPH (as Motor Oil)	3	mg/kg			0									
TPH (as Hydraulic Oil)	3	mg/kg			0									
TPH (as Crude Oil)	3	mg/kg			0									
TPH (as Fuel Oil #6)	3	mg/kg			0									
TPH (as Mineral Spirits)	3	mg/kg			0									
TPH (as Kerosene)	3	mg/kg			0									
TPH (as Jet Fuel JP-4)	3	mg/kg			0									

Target Analyte List: Tampa: PAH (w/methyl naphthalenes) Standard List	Matrix: SOLID Extraction: SOXHLET (NONE,Na2SO4) Method: Hydrocarbons, Polynuclear Aromatic (HPLC - 8310) QC Program: STANDARD TEST SET Location: Quanterra - North Canton
---	---

Compound	RL	Detection Limits			Run Date	Check List 1830					Spike List 1831				
		Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	100	ug/kg	50	ug/kg	19980720	333	ug/kg	15	105	45	333	ug/kg	10	124	50
Acenaphthylene	100	ug/kg	36	ug/kg	19980720	333	ug/kg	10	101	47	333	ug/kg	10	130	50
Anthracene	100	ug/kg	26	ug/kg	19980720	66.7	ug/kg	37	130	47	66.7	ug/kg	10	126	50
Benzo(a)anthracene	5.0	ug/kg	3.5	ug/kg	19980720	66.7	ug/kg	52	105	27	66.7	ug/kg	12	135	50
Benzo(b)fluoranthene	5.0	ug/kg	2.6	ug/kg	19980720	66.7	ug/kg	46	118	36	66.7	ug/kg	10	150	50
Benzo(k)fluoranthene	1.7	ug/kg	0.96	ug/kg	19980720	66.7	ug/kg	48	114	33	66.7	ug/kg	10	159	50
Benzo(ghi)perylene	10.0	ug/kg	6.5	ug/kg	19980722	66.7	ug/kg	37	127	45	66.7	ug/kg	10	116	50
Benzo(a)pyrene	5.0	ug/kg	2.1	ug/kg	19980720	66.7	ug/kg	23	126	52	66.7	ug/kg	10	128	50
Chrysene	5.0	ug/kg	3.1	ug/kg	19980720	66.7	ug/kg	51	107	28	66.7	ug/kg	10	199	50
Dibenz(a,h)anthracene	5.0	ug/kg	1.9	ug/kg	19980720	66.7	ug/kg	43	125	41	66.7	ug/kg	10	110	50
Fluoranthene	10	ug/kg	5.3	ug/kg	19980720	66.7	ug/kg	46	103	29	66.7	ug/kg	14	123	50
Fluorene	100	ug/kg	40	ug/kg	19980720	66.7	ug/kg	21	104	42	66.7	ug/kg	10	142	50
Indeno(1,2,3-cd)pyrene	5.0	ug/kg	4.2	ug/kg	19980722	66.7	ug/kg	41	121	40	66.7	ug/kg	10	116	50
2-Methylnaphthalene	100	ug/kg	38	ug/kg	19980720										
1-Methylnaphthalene	100	ug/kg	37	ug/kg	19980720										
Naphthalene	100	ug/kg	40	ug/kg	19980720	333	ug/kg	10	99	48	333	ug/kg	10	122	50
Phenanthrene	100	ug/kg	53	ug/kg	19980720	66.7	ug/kg	36	113	39	333	ug/kg	10	155	50
Pyrene	5.0	ug/kg	4.8	ug/kg	19980720	66.7	ug/kg	51	108	29	66.7	ug/kg	10	140	50
Benzo(e)pyrene						66.7	ug/kg	25	176	0	66.7	ug/kg	25	176	0
Terphenyl-d14						66.7	ug/kg	10	141	0	66.7	ug/kg	10	141	0

Quanterra Reference Data Summary

Target Analyte List: Q: SW846 Method 8021 Standard List

Matrix: WATER
 Extraction: PURGE AND TRAP - 5 mL purge
 Method: Volatile Organics (8021B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2313		Detection Limits				Check List 914					Spike List 915				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Benzene	1	ug/L	0.013	ug/L	19980219			73	134	20			55	161	25
Bromobenzene	1	ug/L	0.18	ug/L	19980225										
Bromochloromethane	1	ug/L	0.23	ug/L	19980225										
Bromodichloromethane	1	ug/L	0.56	ug/L	19980218										
Bromoform	1	ug/L	0.4	ug/L	19980218										
Bromomethane	1	ug/L	0.29	ug/L	19980225										
n-Butylbenzene	1	ug/L			0										
sec-Butylbenzene	1	ug/L			0										
tert-Butylbenzene	1	ug/L			0										
Carbon tetrachloride	1	ug/L	0.59	ug/L	19980218										
Chlorobenzene	1	ug/L	0.51	ug/L	19980218			61	134	20			42	147	23
Chlorodibromomethane	1	ug/L	0.34	ug/L	19980218										
Chloroethane	1	ug/L	0.5	ug/L	19980218										
Chloroform	1	ug/L	0.37	ug/L	19980218										
Chloromethane	1	ug/L	0.4	ug/L	19980218										
2-Chlorotoluene	1	ug/L	0.39	ug/L	19980218										
4-Chlorotoluene	1	ug/L	0.4	ug/L	19980218										
1,2-Dibromo-3-chloropropane (DBCP)	1	ug/L	0.6	ug/L	19980218										
1,2-Dibromoethane (EDB)	1	ug/L	0.43	ug/L	19980218										
Dibromomethane	1	ug/L	0.42	ug/L	19980218										
1,2-Dichlorobenzene	1	ug/L	0.38	ug/L	19980218										
1,3-Dichlorobenzene	1	ug/L	0.26	ug/L	19980218										
1,4-Dichlorobenzene	1	ug/L	0.45	ug/L	19980218										
Dichlorodifluoromethane	1	ug/L	0.26	ug/L	19980218										
1,1-Dichloroethane	1	ug/L	0.38	ug/L	19980218										
1,2-Dichloroethane	1	ug/L	0.37	ug/L	19980218										
cis-1,2-Dichloroethene	1	ug/L	0.78	ug/L	19980218										
trans-1,2-Dichloroethene	1	ug/L	0.41	ug/L	19980218										
1,1-Dichloroethene	1	ug/L	0.27	ug/L	19980218			35	127	20			14	151	28
1,2-Dichloropropane	1	ug/L	0.52	ug/L	19980218										
1,3-Dichloropropane	1	ug/L	0.18	ug/L	19980225										
2,2-Dichloropropane	1	ug/L	0.78	ug/L	19980218										
cis-1,3-Dichloropropene	1	ug/L	0.6	ug/L	19980313										
trans-1,3-Dichloropropene	1	ug/L	0.32	ug/L	19980218										
1,1-Dichloropropene	1	ug/L	0.59	ug/L	19980218										
Ethylbenzene	1	ug/L	0.026	ug/L	19980219										

Target Analyte List: Q: SW846 Method 8021 Standard List

Matrix: WATER
 Extraction: PURGE AND TRAP - 5 mL purge
 Method: Volatile Organics (8021B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2313		Detection Limits			Run Date	Check List 914			Spike List 915		
Compound	RL	Units	MDL	Units		Amt	Units	LCL UCL RPD	Amt	Units	LCL UCL RPD
Hexachlorobutadiene	1	ug/L	0.25	ug/L	19980225						
Isopropylbenzene	1	ug/L			0						
p-Isopropyltoluene	1	ug/L			0						
Methylene chloride	5	ug/L	0.36	ug/L	19980218						
Naphthalene	1	ug/L	0.54	ug/L	19980219						
n-Propylbenzene	1	ug/L			0						
Styrene	1	ug/L			0						
1,1,1,2-Tetrachloroethane	1	ug/L	0.65	ug/L	19980218						
1,1,2,2-Tetrachloroethane	1	ug/L	0.32	ug/L	19980225						
Tetrachloroethene	1	ug/L	0.2	ug/L	19980225						
Toluene	1	ug/L	0.024	ug/L	19980219			71 132 20			55 159 25
1,2,3-Trichlorobenzene	1	ug/L	0.21	ug/L	19980218						
1,2,4-Trichlorobenzene	1	ug/L	0.57	ug/L	19980218						
1,1,1-Trichloroethane	1	ug/L	0.37	ug/L	19980218						
1,1,2-Trichloroethane	1	ug/L	0.4	ug/L	19980218						
Trichloroethene	1	ug/L	0.41	ug/L	19980218			58 131 20			10 229 41
Trichlorofluoromethane	1	ug/L	0.39	ug/L	19980218						
1,2,3-Trichloropropane	1	ug/L	0.73	ug/L	19980218						
1,2,4-Trimethylbenzene	1	ug/L	0.054	ug/L	19980219						
1,3,5-Trimethylbenzene	1	ug/L	0.04	ug/L	19980219						
Vinyl chloride	1	ug/L	0.82	ug/L	19980218						
Xylenes (total)	1	ug/L	0.3	ug/L	19990427						
1,4-Dichlorobutane								50 150 0			50 150 0
Trifluorotoluene								50 150 0			50 150 0

Quanterra Report Data Summary

Target Analyte List: Q: CLP MSSemi TCL Standard List

Matrix: WATER
 Extraction: LIQ/LIQ, CONT (A/B/N) - Acid->Base
 Method: Base/Neutrals and Acids (8270C)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2101		Detection Limits		Check List 1660					Spike List 1661						
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	10	ug/L	2.7	ug/L	19980305	50	ug/L	47	145	26	50	ug/L	47	145	28
Acenaphthylene	10	ug/L	2.8	ug/L	19980305										
Anthracene	10	ug/L	2.7	ug/L	19980305										
Benzo(a)anthracene	10	ug/L	2.7	ug/L	19980305										
Benzo(b)fluoranthene	10	ug/L	2.7	ug/L	19980305										
Benzo(k)fluoranthene	10	ug/L	3.0	ug/L	19980305										
Benzo(ghi)perylene	10	ug/L	3.2	ug/L	19980305										
Benzo(a)pyrene	10	ug/L	2.7	ug/L	19980305										
bis(2-Chloroethoxy)methane	10	ug/L	2.6	ug/L	19980305										
bis(2-Chloroethyl) ether	10	ug/L	2.8	ug/L	19980305										
bis(2-Ethylhexyl) phthalate	10	ug/L	3.0	ug/L	19980305										
4-Bromophenyl phenyl ether	10	ug/L	2.7	ug/L	19980305										
Butyl benzyl phthalate	10	ug/L	2.3	ug/L	19980305										
Carbazole	10	ug/L	2.8	ug/L	19980305										
4-Chloroaniline	10	ug/L	3.8	ug/L	19980305										
4-Chloro-3-methylphenol	10	ug/L	2.9	ug/L	19980305	75	ug/L	22	147	31	75	ug/L	22	147	37
2-Chloronaphthalene	10	ug/L	2.5	ug/L	19980305										
2-Chlorophenol	10	ug/L	3.0	ug/L	19980305	75	ug/L	23	134	47	75	ug/L	23	134	29
4-Chlorophenyl phenyl ether	10	ug/L	2.8	ug/L	19980305										
Chrysene	10	ug/L	3.0	ug/L	19980305										
Dibenz(a,h)anthracene	10	ug/L	3.2	ug/L	19980305										
Dibenzofuran	10	ug/L	2.9	ug/L	19980305										
Di-n-butyl phthalate	10	ug/L	2.8	ug/L	19980305										
1,2-Dichlorobenzene	10	ug/L	2.5	ug/L	19980305										
1,3-Dichlorobenzene	10	ug/L	2.6	ug/L	19980305										
1,4-Dichlorobenzene	10	ug/L	2.6	ug/L	19980305	50	ug/L	20	124	25	50	ug/L	20	124	32
3,3'-Dichlorobenzidine	50	ug/L	2.7	ug/L	19980305										
2,4-Dichlorophenol	10	ug/L	2.9	ug/L	19980305										
Diethyl phthalate	10	ug/L	2.4	ug/L	19980305										
2,4-Dimethylphenol	10	ug/L	2.8	ug/L	19980305										
Dimethyl phthalate	10	ug/L	2.6	ug/L	19980305										
4,6-Dinitro-2-methylphenol	50	ug/L	3.4	ug/L	19980305										
2,4-Dinitrophenol	50	ug/L	3.3	ug/L	19980305										
2,4-Dinitrotoluene	10	ug/L	3.1	ug/L	19980305	50	ug/L	60	134	37	50	ug/L	39	139	22
2,6-Dinitrotoluene	10	ug/L	2.7	ug/L	19980305										
Di-n-octyl phthalate	10	ug/L	3.1	ug/L	19980305										

Target Analyte List: Q: CLP MSSemi TCL Standard List

Matrix: WATER
 Extraction: LIQ/LIQ, CONT (A/B/N) - Acid->Base
 Method: Base/Neutrals and Acids (8270C)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2101		Detection Limits				Check List 1660					Spike List 1661				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Fluoranthene	10	ug/L	3.2	ug/L	19980305										
Fluorene	10	ug/L	2.7	ug/L	19980305										
Hexachlorobenzene	10	ug/L	2.9	ug/L	19980305										
Hexachlorobutadiene	10	ug/L	2.6	ug/L	19980203										
Hexachlorocyclopentadiene	50	ug/L	1	ug/L	19980304										
Hexachloroethane	10	ug/L	2.4	ug/L	19980305										
Indeno(1,2,3-cd)pyrene	10	ug/L	2.9	ug/L	19980305										
Isophorone	10	ug/L	2.8	ug/L	19980305										
2-Methylnaphthalene	10	ug/L	3	ug/L	19980305										
2-Methylphenol	10	ug/L	2.9	ug/L	19980305										
4-Methylphenol	10	ug/L	3.1	ug/L	19980305										
Naphthalene	10	ug/L	2.7	ug/L	19980305										
2-Nitroaniline	50	ug/L	3.3	ug/L	19980305										
3-Nitroaniline	50	ug/L	3	ug/L	19980305										
4-Nitroaniline	50	ug/L	2.8	ug/L	19980305										
Nitrobenzene	10	ug/L	2.9	ug/L	19980305										
2-Nitrophenol	10	ug/L	2.9	ug/L	19980305										
4-Nitrophenol	50	ug/L	3.4	ug/L	19980305	75	ug/L	30	162	47	75	ug/L	10	132	47
N-Nitrosodiphenylamine	10	ug/L	2.9	ug/L	19980305										
N-Nitrosodi-n-propylamine	10	ug/L	2.7	ug/L	19980305	50	ug/L	10	230	33	50	ug/L	10	230	55
Pentachlorophenol	10	ug/L	3.3	ug/L	19980305	75	ug/L	14	176	47	75	ug/L	14	176	49
Phenanthrene	10	ug/L	2.9	ug/L	19980305										
Phenol	10	ug/L	2.7	ug/L	19980305	75	ug/L	10	112	49	75	ug/L	10	112	23
2,2'-Oxybis(1-Chloropropane)	10	ug/L	3.2	ug/L	19980305										
Pyrene	10	ug/L	2.8	ug/L	19980305	50	ug/L	68	131	45	50	ug/L	52	115	25
1,2,4-Trichlorobenzene	10	ug/L	2.5	ug/L	19980305	50	ug/L	44	142	25	50	ug/L	44	142	28
2,4,5-Trichlorophenol	10	ug/L	3.1	ug/L	19980305										
2,4,6-Trichlorophenol	10	ug/L	2.9	ug/L	19980305										
2-Fluorobiphenyl						50	ug/L	45	118	0	50	ug/L	45	118	0
2-Fluorophenol						75	ug/L	21	100	0	75	ug/L	21	100	0
2,4,6-Tribromophenol						75	ug/L	16	129	0	75	ug/L	16	129	0
Nitrobenzene-d5						50	ug/L	40	114	0	50	ug/L	40	114	0
Phenol-d5						75	ug/L	17	101	0	75	ug/L	17	101	0
Terphenyl-d14						50	ug/L	33	141	0	50	ug/L	33	141	0

Quanterra Reference Data Summary

Target Analyte List: Q: CLP MSVOA TCL Standard List

Matrix: WATER
 Extraction: PURGE AND TRAP - 5 mL purge
 Method: Volatile Organics, GC/MS (8260B)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 2001		Detection Limits				Check List 902					Spike List 903				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acetone	20	ug/L	5.9	ug/L	19980213										
Benzene	5	ug/L	0.45	ug/L	19980213	20	ug/L	83	110	13	50	ug/L	78	117	17
Bromodichloromethane	5	ug/L	0.39	ug/L	19980213										
Bromoform	5	ug/L	0.35	ug/L	19980213										
Bromomethane	10	ug/L	1.1	ug/L	19980128										
2-Butanone	20	ug/L	7.6	ug/L	19980213										
Carbon disulfide	5	ug/L	0.4	ug/L	19980213										
Carbon tetrachloride	5	ug/L	0.41	ug/L	19980213										
Chlorobenzene	5	ug/L	0.43	ug/L	19980213	20	ug/L	85	115	15	50	ug/L	81	115	18
Dibromochloromethane	5	ug/L	0.36	ug/L	19980213										
Chloroethane	10	ug/L	0.67	ug/L	19980213										
Chloroform	5	ug/L	0.51	ug/L	19980213										
Chloromethane	10	ug/L	1.1	ug/L	19980213										
1,1-Dichloroethane	5	ug/L	0.62	ug/L	19980213										
1,2-Dichloroethane	5	ug/L	0.43	ug/L	19980213										
1,1-Dichloroethene	5	ug/L	0.53	ug/L	19980213	20	ug/L	70	122	26	50	ug/L	75	113	20
1,2-Dichloroethene (total)	5	ug/L	0.87	ug/L	19980213										
1,2-Dichloropropane	5	ug/L	0.32	ug/L	19980213										
cis-1,3-Dichloropropene	5	ug/L	0.35	ug/L	19980213										
trans-1,3-Dichloropropene	5	ug/L	0.64	ug/L	19980213										
Ethylbenzene	5	ug/L	0.41	ug/L	19980213										
2-Hexanone	20	ug/L	8.6	ug/L	19980213										
Methylene chloride	5	ug/L	0.4	ug/L	19980213										
4-Methyl-2-pentanone	20	ug/L	1.4	ug/L	19980213										
Styrene	5	ug/L	0.43	ug/L	19980213										
1,1,2,2-Tetrachloroethane	5	ug/L	0.57	ug/L	19980213										
Tetrachloroethene	5	ug/L	0.47	ug/L	19980213										
Toluene	5	ug/L	0.45	ug/L	19980213	20	ug/L	86	119	16	50	ug/L	78	126	24
1,1,1-Trichloroethane	5	ug/L	0.63	ug/L	19980213										
1,1,2-Trichloroethane	5	ug/L	0.41	ug/L	19980213										
Trichloroethene	5	ug/L	0.54	ug/L	19980213	20	ug/L	82	112	15	50	ug/L	71	110	22
Vinyl chloride	10	ug/L	0.58	ug/L	19980213										
Xylenes (total)	5	ug/L	1.4	ug/L	19980213										
Bromofluorobenzene						50	ug/L	86	115	0	50	ug/L	86	115	0
1,2-Dichloroethane-d4						50	ug/L	80	120	0	50	ug/L	80	120	0
Toluene-d8						50	ug/L	88	110	0	50	ug/L	88	110	0

Target Analyte List: Q: CLP MSVOA TCL Standard List

Matrix: WATER
Extraction: PURGE AND TRAP - 5 mL purge
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: Quanterra - North Canton

Target List 2001		Detection Limits				Check List 902					Spike List 903				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Dibromofluoromethane						50	ug/L	86	118	0	50	ug/L	86	118	0

Quanterra REPORT Data Summary

Target Analyte List: NC: TCL Pesticide 8081A List

Matrix: WATER
 Extraction: LIQ/LIQ, CONT (PAH,P/P,TPH) - Nominal
 Method: Pesticides (8081A)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Target List 4115		Detection Limits				Check List 912					Spike List 913				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aldrin	0.05	ug/L	0.00450	ug/L	19980130	0.2	ug/L	60	117	29	0.2	ug/L	54	120	40
alpha-BHC	0.05	ug/L	0.007	ug/L	19980218										
beta-BHC	0.05	ug/L	0.011	ug/L	19980221										
delta-BHC	0.05	ug/L	0.00820	ug/L	19980221										
gamma-BHC (Lindane)	0.05	ug/L	0.0069	ug/L	19980218										
alpha-Chlordane	0.05	ug/L	0.016	ug/L	19980221										
gamma-Chlordane	0.05	ug/L	0.0045	ug/L	19980130										
4,4'-DDD	0.05	ug/L	0.0096	ug/L	19980218										
4,4'-DDE	0.05	ug/L	0.008	ug/L	19980303										
4,4'-DDT	0.05	ug/L	0.005	ug/L	19980218	0.5	ug/L	55	128	36	0.5	ug/L	48	154	47
Dieldrin	0.05	ug/L	0.0075	ug/L	19980218	0.5	ug/L	63	122	25	0.5	ug/L	54	143	32
Endosulfan I	0.05	ug/L	0.0072	ug/L	19980218										
Endosulfan II	0.05	ug/L	0.0078	ug/L	19980303										
Endosulfan sulfate	0.05	ug/L	0.011	ug/L	19980221										
Endrin	0.05	ug/L	0.013	ug/L	19980218	0.5	ug/L	48	129	41	0.5	ug/L	64	142	39
Endrin aldehyde	0.05	ug/L	0.0097	ug/L	19980221										
Endrin ketone	0.05	ug/L	0.02	ug/L	19980221										
Heptachlor	0.05	ug/L	0.0025	ug/L	19980218	0.2	ug/L	56	125	34	0.2	ug/L	56	158	36
Heptachlor epoxide	0.05	ug/L	0.0092	ug/L	19980221										
Methoxychlor	0.1	ug/L	0.049	ug/L	19980218										
Toxaphene	2	ug/L	0.33	ug/L	19980221										
Decachlorobiphenyl						0.2	ug/L	10	116	0	0.2	ug/L	10	116	0
Tetrachloro-m-xylene						0.2	ug/L	10	130	0	0.2	ug/L	10	130	0

Quanterra Reference Data Summary

Target Analyte List: Q: Aroclors Only Standard List	Matrix:	WATER
	Extraction:	LIQ/LIQ, CONT w/ACID STRIP (PCB) - Nominal
	Method:	PCBs (8082)
	QC Program:	STANDARD TEST SET
	Location:	Quanterra - North Canton

Target List 2207		Detection Limits			Check List 1832					Spike List 1833					
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aroclor 1016	1	ug/L	0.41	ug/L	19980319	10	ug/L	66	111	23	10	ug/L	42	136	29
Aroclor 1221	1	ug/L	0.47	ug/L	19980319										
Aroclor 1232	1	ug/L	0.12	ug/L	19980202										
Aroclor 1242	1	ug/L	0.47	ug/L	19980319										
Aroclor 1248	1	ug/L	0.37	ug/L	19980319										
Aroclor 1254	1	ug/L	0.21	ug/L	19980319										
Aroclor 1260	1	ug/L	0.36	ug/L	19980516	10	ug/L	65	111	23	10	ug/L	42	136	29
Decachlorobiphenyl						0.2	ug/L	10	116	0	0.2	ug/L	10	116	0
Tetrachloro-m-xylene						0.2	ug/L	10	130	0	0.2	ug/L	10	130	0

Quanterra Reference Data Summary

Target Analyte List: All Analytes	Matrix: WATER Extraction: LIQ/LIQ, CONT (PAH,P/P,TPH) - Nominal Method: Hydrocarbons, Extractable Petroleum (8015B) QC Program: STANDARD TEST SET Location: Quanterra - North Canton
-----------------------------------	--

Analyte List		Detection Limits			Check List 1801					Spike List 1802					
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
TPH (Extractables)	100	ug/L			0										
Total Petroleum Hydrocarbons - Extrac	100	ug/L			0			17	110	77			10	116	77
Total Petroleum Hydrocarbons	100	ug/L			0										
TPH (as Diesel)	100	ug/L			0										
TPH (as Motor Oil)	300	ug/L			0										
TPH (as Hydraulic Oil)	100	ug/L			0										
TPH (as Crude Oil)	100	ug/L			0										
TPH (as Fuel Oil #6)	100	ug/L			0										
TPH (as Mineral Spirits)	100	ug/L			0										
TPH (as Kerosene)	100	ug/L			0										
TPH (as Jet Fuel JP-4)	100	ug/L			0										

Target Analyte List: All Analytes

Matrix: WATER
Extraction: PURGE AND TRAP - 5 mL purge
Method: Hydrocarbons, Volatile Petroleum (8015B)
QC Program: STANDARD TEST SET
Location: Quanterra - North Canton

Analyte List Compound	RL	Detection Limits			Run Date	Check List 1600			Spike List 1601				
		Units	MDL	Units		Amt	Units	LCL UCL RPD	Amt	Units	LCL UCL RPD		
TPH (as Gasoline)	100	ug/L			0		70	130	77		10	116	77

Quanterra K101000 Data Summary

Target Analyte List: Tampa: PAH (w/methyl naphthalenes) Standard List

Matrix: WATER
 Extraction: LIQ/LIQ, SEP FUNNEL (PAH,P/P,TPH,Dioxin) - Nominal
 Method: Hydrocarbons, Polynuclear Aromatic (HPLC - 8310)
 QC Program: STANDARD TEST SET
 Location: Quanterra - North Canton

Compound	RL	Detection Limits			Run Date	Check List 1830					Spike List 1831				
		Units	MDL	Units		Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	2 4.0	ug/L	0.85	ug/L	19980604	10	ug/L	10	101	48	10	ug/L	10	124	50
Acenaphthylene	2 4.0	ug/L	0.64	ug/L	19980604	10	ug/L	10	99	49	10	ug/L	10	139	50
Anthracene	1 2	ug/L	1.2	ug/L	19980604	2	ug/L	18	126	59	2	ug/L	10	126	50
Benzo(a)anthracene	.13 0.40	ug/L	0.096	ug/L	19980604	2	ug/L	44	116	36	2	ug/L	12	135	50
Benzo(b)fluoranthene	.18 0.10	ug/L	0.098	ug/L	19980604	2	ug/L	39	125	43	2	ug/L	10	150	50
Benzo(k)fluoranthene	.17 0.05	ug/L	0.047	ug/L	19980604	2	ug/L	38	124	43	2	ug/L	10	159	50
Benzo(ghi)perylene	.2 0.1	ug/L	0.1	ug/L	19980604	2	ug/L	23	116	54	2	ug/L	10	116	50
Benzo(a)pyrene	0.2 0.1	ug/L	0.091	ug/L	19980604	2	ug/L	22	128	64	2	ug/L	10	128	50
Chrysene	.2 0.1	ug/L	0.094	ug/L	19980604	2	ug/L	38	118	40	2	ug/L	10	199	50
Dibenz(a,h)anthracene	.2 0.1	ug/L	0.096	ug/L	19980604	2	ug/L	22	110	54	2	ug/L	10	110	50
Fluoranthene	.9 0.1	ug/L	0.098	ug/L	19980604	2	ug/L	43	102	29	2	ug/L	14	123	50
Fluorene	1 1	ug/L	0.68	ug/L	19980604	2	ug/L	13	100	43	2	ug/L	10	142	50
Indeno(1,2,3-cd)pyrene	.2 0.1	ug/L	0.093	ug/L	19980604	2	ug/L	36	116	44	2	ug/L	10	116	50
2-Methylnaphthalene	2	ug/L	0.85	ug/L	19980604										
1-Methylnaphthalene	2	ug/L	0.71	ug/L	19980604										
Naphthalene	2	ug/L	1.2	ug/L	19980604	10	ug/L	10	90	46	10	ug/L	10	122	50
Phenanthrene	1	ug/L	0.68	ug/L	19980604	2	ug/L	28	113	43	2	ug/L	10	155	50
Pyrene	.5 0.1	ug/L	0.086	ug/L	19980604	2	ug/L	38	118	40	2	ug/L	10	140	50
Benzo(e)pyrene						2	ug/L	39	182	0	2	ug/L	39	182	0
Terphenyl-d14						2	ug/L	33	120	0	2	ug/L	33	120	0

QUANTERRA REFERENCE DATA SUMMARY

Target Analyte List: KNX: HPLC 8330 STANDARD LIST

Matrix: WATER
 Extraction: EXTRACTION, SOLID PHASE
 Method: Nitroaromatics & Nitramines "Explosives" (8330)
 QC Program: STANDARD TEST SET
 Location: Quanterra - Knoxville

Target List 4832		Detection Limits				Check List 8215					Spike List 8216				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
4-Amino-2,6-dinitrotoluene	0.2	ug/L	0.04	ug/L	19980128	3.0	ug/L	66	115	27	3.0	ug/L	37	148	27
2-Amino-4,6-dinitrotoluene	0.2	ug/L	0.04	ug/L	19980128	3.0	ug/L	67	115	29	3.0	ug/L	44	153	29
1,3-Dinitrobenzene	0.2	ug/L	0.03	ug/L	19980128	3.0	ug/L	75	121	27	3.0	ug/L	56	155	27
2,4-Dinitrotoluene	0.2	ug/L	0.03	ug/L	19980128	3.0	ug/L	73	118	30	3.0	ug/L	56	147	30
2,6-Dinitrotoluene	0.2	ug/L	0.03	ug/L	19980128	3.0	ug/L	71	123	31	3.0	ug/L	52	149	31
HMX	0.5	ug/L	0.06	ug/L	19980128	3.0	ug/L	70	125	28	3.0	ug/L	57	131	28
Nitrobenzene	0.2	ug/L	0.04	ug/L	19980128	3.0	ug/L	62	115	28	3.0	ug/L	35	150	28
2-Nitrotoluene	0.2	ug/L	0.05	ug/L	19980128	3.0	ug/L	52	115	30	3.0	ug/L	30	148	30
4-Nitrotoluene	0.2	ug/L	0.07	ug/L	19980128	3.0	ug/L	57	115	33	3.0	ug/L	31	155	33
3-Nitrotoluene	0.2	ug/L	0.05	ug/L	19980128	3.0	ug/L	57	115	35	3.0	ug/L	30	156	35
RDX	0.5	ug/L	0.04	ug/L	19980128	3.0	ug/L	78	125	24	3.0	ug/L	56	163	24
Tetryl	0.2	ug/L	0.03	ug/L	19980128	3.0	ug/L	72	125	27	3.0	ug/L	48	133	27
1,3,5-Trinitrobenzene	0.2	ug/L	0.03	ug/L	19980128	3.0	ug/L	76	120	30	3.0	ug/L	50	159	30
2,4,6-Trinitrotoluene	0.2	ug/L	0.06	ug/L	19980128	3.0	ug/L	73	122	27	3.0	ug/L	55	137	27
1-Chloro-3-nitrobenzene						1.77	ug/L	32	127	0	1.77	ug/L	32	127	0

Quanterra Reference Data Summary

Target Analyte List: All Analytes	Matrix: SOLID Extraction: None specified. Method: Carbon, Total Organic "TOC" (WALKLEY) - Solids QC Program: STANDARD TEST SET Location: Quanterra - North Canton
-----------------------------------	---

Analyte List	Detection Limits					Check List 1215					Spike List 1216				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Total Organic Carbon	100	mg/kg			0	3750	mg/kg	69	130	20	611500	mg/kg	75	125	20

Quanterra Reference Data Summary

Target Analyte List: All Analytes	Matrix: WATER Extraction: None specified. Method: Carbon, Total Organic "TOC" (415.1) QC Program: STANDARD TEST SET Location: Quanterra - North Canton
-----------------------------------	--

Analyte List	Detection Limits					Check List 1215					Spike List 1216				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Total Organic Carbon	1.0	mg/L	0.47	mg/L	19980228	10	mg/L	90	110	20	25	mg/L	90	110	20

Quanterra Reference Data Summary

Target Analyte List: All Analytes	Matrix: WATER Extraction: METALS, TOTAL RECOVERABLE Method: Inductively Coupled Plasma (6010B Trace) QC Program: STANDARD TEST SET Location: Quanterra - North Canton
-----------------------------------	---

Analyte List		Detection Limits				Check List 904					Spike List 905				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Antimony	0.01	mg/L	3.4	ug/L	19980204	0.5	mg/L	80	120	20	0.5	mg/L	80	120	20
Arsenic	0.01	mg/L	3	ug/L	19980204	2	mg/L	80	120	20	2	mg/L	80	120	20
Cadmium	0.002	mg/L	0.62	ug/L	19980224	0.05	mg/L	80	120	20	0.05	mg/L	80	120	20
Chromium	0.005	mg/L	3.4	ug/L	19980225	0.2	mg/L	80	120	20	0.2	mg/L	80	120	20
Cobalt	0.007	mg/L	1.4	ug/L	19980224	0.5	mg/L	80	120	20	0.5	mg/L	80	120	20
Lead	0.003	mg/L	2.1	ug/L	19980224	0.5	mg/L	80	120	20	0.5	mg/L	80	120	20
Molybdenum	0.01	mg/L	2.7	ug/L	19980204	1	mg/L	80	120	20	1	mg/L	80	120	20
Selenium	0.005	mg/L	3.9	ug/L	19980204	2	mg/L	80	120	20	2	mg/L	80	120	20
Silver	0.005	mg/L	0.59	ug/L	19980204	0.05	mg/L	80	120	20	0.05	mg/L	80	120	20
Thallium	0.010	mg/L	9.7	ug/L	19980225	2	mg/L	80	120	20	2	mg/L	80	120	20
Vanadium	0.007	mg/L	1	ug/L	19980225	0.5	mg/L	80	120	20	0.5	mg/L	80	120	20

Quanterra Reference Data Summary

Target Analyte List: Q: CLP ICP Metals TAL Standard List	Matrix: WATER Extraction: METALS, TOTAL RECOVERABLE Method: Inductively Coupled Plasma (6010B) QC Program: STANDARD TEST SET Location: Quanterra - North Canton
--	---

Target List 2600			Detection Limits			Check List 906					Spike List 907				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	0.2	mg/L	54	ug/L	19980204	2.0	mg/L	87	115	14	2.0	mg/L	80	120	20
Antimony	0.06	mg/L	25	ug/L	19980204	0.50	mg/L	87	108	11	0.50	mg/L	80	120	20
Barium	0.20	mg/L	3.4	ug/L	19980204	2.0	mg/L	87	110	12	2.0	mg/L	80	120	20
Beryllium	0.005	mg/L	0.67	ug/L	19980204	0.050	mg/L	85	110	12	0.050	mg/L	80	120	20
Cadmium	0.005	mg/L	3.5	ug/L	19980204	0.050	mg/L	89	115	13	0.050	mg/L	80	120	20
Calcium	5.0	mg/l.	150	ug/L	19980204	50	mg/L	86	109	12	50	mg/L	80	120	20
Chromium	0.01	mg/L	7	ug/L	19980204	0.20	mg/L	86	112	13	0.20	mg/L	80	120	20
Cobalt	0.05	mg/L	17	ug/L	19980204	0.50	mg/L	83	107	12	0.50	mg/L	80	120	20
Copper	0.025	mg/L	3.1	ug/L	19980204	0.25	mg/L	84	112	14	0.25	mg/L	80	120	20
Iron	0.1	mg/L	50	ug/L	19980325	1.0	mg/L	80	120	20	1.0	mg/L	80	120	20
Magnesium	5.0	mg/L	52	ug/L	19980204	50	mg/L	88	112	12	50	mg/L	80	120	20
Manganese	0.015	mg/L	3.1	ug/l.	19980204	0.50	mg/L	88	117	14	0.50	mg/L	80	120	20
Nickel	0.04	mg/L	15	ug/L	19980204	0.50	mg/L	85	116	15	0.50	mg/L	80	120	20
Potassium	5	mg/L	120	ug/L	19980204	50	mg/L	87	106	9	50	mg/L	80	120	20
Silver	0.01	mg/L	3	ug/L	19980204	0.05	mg/L	93	120	14	0.05	mg/L	80	120	20
Sodium	5	mg/L	340	ug/L	19980204	50	mg/L	88	107	9	50	mg/L	80	120	20
Vanadium	0.05	mg/L	6.5	ug/L	19980204	0.50	mg/L	86	111	12	0.50	mg/L	80	120	20
Zinc	0.02	mg/L	11	ug/L	19980204	0.50	mg/L	83	120	24	0.50	mg/L	80	120	20

28 October 1998

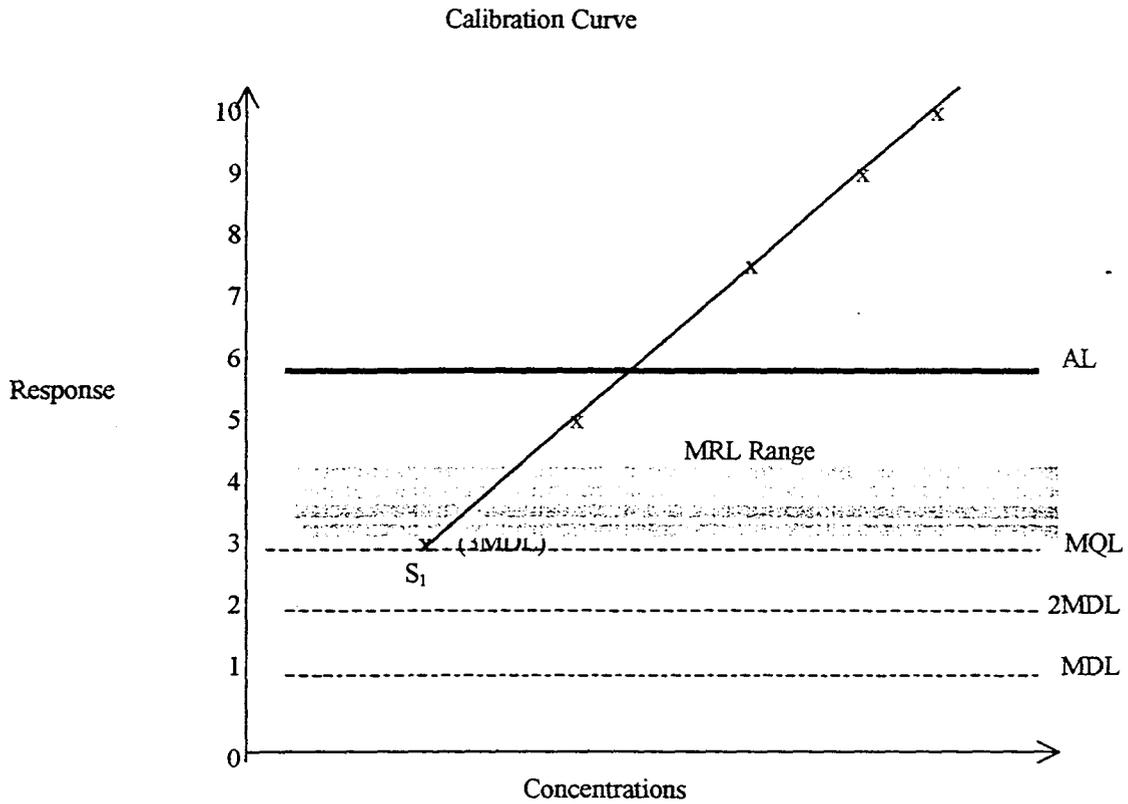
MEMORANDUM FOR Record

SUBJECT: Method Reporting Limit

1. This memorandum summarizes the various detection limits and outlines a procedure for establishing the reporting limit for projects.
2. MDL: Method Detection Limits are measured by multiplying the student t with the standard deviation (SD) of seven replicates i.e. the MDL is the minimum concentration of a substance that can be measured/detected above zero at 99% confidence in a given matrix. MDLs are determined according to 40 CFR, Part 136, Appendix B or any other statistical methods. MDL check sample must be performed and be detected at two times the MDL.
3. MQL: Method Quantitation Limits are established at 3-10 times the MDL levels. The MQLs that were established at such high levels due to the inherited error ($\pm 100\%$) associated with the results calculated at the MDL. The error associated with the MQL should be comparable to the CCV acceptance limits ($\pm 15\%$), the statistical error is notably reduced from that of the MDL. Consequently the MDL must be set at the **lowest concentration standard** used for the initial calibration curve. Also, the lowest standard or low-level calibration verification standard must be at least **three times** the MDL. Based on these criteria, analytes detected below the MQL must be flagged with "J" for estimated quantities.
4. MRL: Method Reporting Limits are threshold values above which results are reported as positive quantities, and below which results are reported as non-detect (<) and flagged with "U", or as estimated and flagged with "J". MRL must not be established at levels below the MDL check sample level. MRL value is dependent upon project specifics as discussed in item 6.
5. Relation of MRLs to Action Levels (ALs): establishing MRL must be based upon data user, data needs, maximum size of the errors that the user is willing to accept, and the capability of the instrument/method. MRL varies according to project goals, and as a general rule the USACE recommends **the MRLs be established at approximately one-half the project action level**. Therefore, MRL may be established within a range from 2MDL to one-half of the action level. The MRL must have a point on the calibration curve.
6. As an example for explosive analyses, MRL must be established between the MQL (3MDL) and the AL. Accordingly, nitroaromatic compounds detected below the

MRL must be reported as non-detect (<), and levels identified below the MRL must not be reported.

7. Compilation of items 2-6 are illustrated on the graph below:



- Response is expressed as multiples of MDL

8. For further discussion, please contact the undersigned at 502-582-6946.

SAMIR A MANSY, Ph.D.
Quality Assurance manager

Comment Responses

Document: Quality Assurance Project Plan (QAPP) , Limited Site Inspection for the Pentolite Area Waste Lagoons.

Name: Ronald E. Nabors, Ohio EPA

1. General Comment: The Ohio EPA, DERR, would prefer to receive the Quality Assurance Project Plan under separate cover and have it referenced in all site investigation Work Plann submittals.

Response: We apologize for not obtaining an OEPA, DERR preferred format to develop projects in Illinois, Michigan and Ohio. The QAPP and associated FSP and DQO attachments were assembled for the Former Plum Brook Ordnance Works using the EPA Region 5 model QAPP, the format we typically use. We apologize for this inconvenience. If our office executes future phases of work for this Formerly Used Defense Site, we will incorporate the OEPA, DERR preferred format in preparing documents.

2. General Comment: The flow of the document is very confusing as it jumps back and forth between the entire Plum Brook Ordnance Works (PBOW) history of the Pentolite Area Waste Lagoons (PAWL). Please create a Plum Brook Ordnance Works general history section and focus the remainder of the document on the Pentolite Area Waste Lagoons site investigation.

Response: The document will be edited accordingly.

3. Section 1.2.1 General, Page 1: Please indicate the approximate distance from the Pentolite Area Waste Lagoons to the nearest residence.

Response: The approximate distance between the nearest residence and the Pentoloite Area Waste Lagoons is $\frac{3}{4}$ of a mile. This information will be added to section 1.2.1 of the document.

4. Section 1.2.3 PAWL, Site Specific, page 2: Please note what landmarks border the PAWL, not the entire PBOW facility.

Response: The former PAWL area is bordered to the north by the PBRF, to the south by Pentolite Road and by access roads to the east and west. This information will be added to Section 1.2.4.

5. Section 1.2.3 PAWL, Site Specific, Page 2: Please change the name of this section to "Site Specific Operational History."

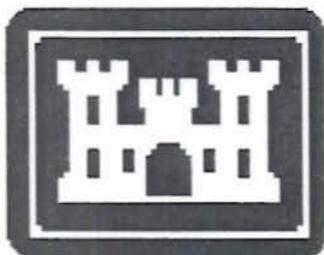
Response: The report will be edited accordingly.

6. Section 1.2.3 PAWL, Site Specific, Page 3: Add a figure that shows the construction design of the waste lagoons discussed in the second paragraph.

Response: Figures will be added to the Figures section, and will be referenced in former

7. Section 1.6.3 Rationale of Selected Sampling Locations, page 9: This section states that the rationale used to select sampling depths will be discussed in the Field Sampling Plan and the Data Quality Objective sections. The rationale for sample locations is not clearly defined in either of these sections. Please give justification for each of the sampling locations and depths chosen.

Response: The rationale for the sampling locations and depth as spelled out in the QAPP, DQOs and FSP was to sample the most likely subsurface material with the potential for contamination. That is, the soils within and below the former lagoon locations. This is stated in section 4.0 of the FSP, Section 3.7 of the DQOs and 1.4 of the QAPP. Specific features of the lagoons, such as inlet and outlet locations and vitreous sewer pipes, were not targeted for this limited SI. Specific features of the lagoons can be targeted for the future sampling events.



**U.S. ARMY CORPS
OF ENGINEERS
LOUISVILLE DISTRICT**

Field Sampling Plan

Limited Site Investigation of the former

Plum Brook Ordnance Works

Pentolite Area Waste Lagoons

Sandusky, Ohio

DERP-FUDS HTRW Project No. G05OH001819

JULY 2000

TABLE OF CONTENTS

1.0	PROJECT DESCRIPTION.....	1
1.1	<i>Introduction</i>	1
1.2	<i>History and Usage</i>	1
	1.2.1 <i>PBOW Facility</i>	1
	1.2.2 <i>Site Specific</i>	2
1.3	<i>Location</i>	4
1.4	<i>Climate</i>	4
1.5	<i>Topography</i>	4
1.6	<i>Geology</i>	4
	1.6.1 <i>PBOW (Installation wide)</i>	4
	1.6.2 <i>PAWL (Site Specific)</i>	5
1.7	<i>Hydrogeology</i>	5
	1.7.1 <i>PBOW (Installation Wide)</i>	5
	1.7.2 <i>PAWL (Site Specific)</i>	6
2.0	PROJECT ORGANIZATION AND RESPONSIBILITIES.....	6
2.1	<i>General</i>	6
2.2	<i>Team Members</i>	6
3.0	SCOPE AND OBJECTIVES.....	7
4.0	SAMPLING DESIGN AND RATIONALE.....	7
5.0	FIELD ACTIVITIES AND SAMPLING PROCEDURES.....	8
5.1	<i>Visual Inspections</i>	8
5.2	<i>Sample Documentation</i>	8

5.3	<i>Photographs</i>	9
5.4	<i>Soil Sampling Procedures</i>	9
5.5	<i>Surface Water and Sediment Sampling</i>	10
5.6	<i>Field Quality Control Sampling</i>	10
5.7	<i>Decontamination</i>	10
6.0	FIELD SAMPLE IDENTIFICATION AND CUSTODY PROCEDURES.....	12
6.1	<i>Sample Containers, Preservation and Holding Times</i>	12
6.2	<i>Sample Identification</i>	14
6.3	<i>Sample Packaging</i>	14
6.4	<i>Custody Transfer and Shipment Procedures</i>	15
7.0	DISPOSITION OF FIELD INVESTIGATION DERIVED WASTE (IDW).....	15
8.0	SCHEDULE.....	16
8.1	<i>Start Date</i>	16
8.2	<i>Pre-mobilization</i>	16
8.3	<i>Mobilization</i>	17
8.4	<i>Demobilization</i>	17

ATTACHMENTS:

1. FIGURES
2. FIELD SCREENING PROCEDURES

1.0 PROJECT DESCRIPTION

1.1 Introduction

Located near Sandusky Ohio, the former Plum Brook Ordnance Works (PBOW) was operated from 1941 to 1945 by an army contractor as a manufacturing plant for trinitrotoluene (TNT), dinitrotoluene (DNT), and pentolite. Contamination previously detected at the site by studies has been related to those activities and is being addressed by the Department of Defense (DOD) under the frame of the Defense Environmental Restoration Program (DERP), Formerly Used Defense Sites (FUDS).

A limited Site Investigation (SI) of the Pentolite Area Waste Lagoons (PAWL), one of the former PBOW sites, will be conducted under this project. The purpose of the SI is to evaluate the potential for contamination of the sites that may be traced to Army activities.

This project is one of several limited SI projects simultaneously undertaken by the U.S. Army Corps of Engineers to be executed by the Louisville District (CELRL) under the direction of the Huntington District (CELRH).

1.2 History and Usage

1.2.1 PBOW Facility

Based on the Archives Search Report (USACE, 1993), the facility was established in 1941 and referred to as Plum Brook Ordnance Works (PBOW). The installation was established for the purpose of manufacturing trinitrotoluene (TNT), dinitrotoluene (DNT), pentolite, and nitric and sulfuric acids. Built by E.B. Badger and Sons Company, the facility was operated under contract by the Trojan Powder Company. Production of explosives ceased two weeks after V-J Day, having manufactured in excess of one billion pounds of explosives during the four-year operating period.

By September 1945, the entire Ordnance Inspection Department was abolished. Decontamination of TNT, acid, pentolite and DNT manufacturing lines was completed during the last quarter of 1945. On 17 December 1945, the physical custody of the plant was transferred from Trojan to the Ordnance Department. The U.S. Army Corps of Engineers assumed responsibility for maintenance and custodial duties until September 1946 when the property was transferred to the War Assets Administration (predecessor to the Government Services Administration (GSA)), after it was certified by the U.S. Army to be decontaminated. National Aeronautical and Space Administration (NASA)

acquired the PBOW in 1963 and is presently using the site, now referred to as Plum Brook Station (PBS).

The PBS site currently lies in an area that is primarily rural and agricultural with a low population density. The NASA Glenn Research Center occupies a majority of the former ordnance works. The Department of the Army maintains a reserve center on the westernmost portion of the facility. The remainder of the former installation is in private ownership with the vast majority being cultivated. A tract on the northern boundary is owned by the Perkins Board of Education and is utilized as a bus maintenance facility

1.2.2 Site Specific

Based on the PBS Preliminary Assessment (PA) (Science Applications International Corporation Report (SAIC), 1991), the first stage of the pentolite manufacturing process involved the nitration of pentaerythritol (PE) by adding nitric acid and water. Pentaerythritol tetranitrate (PETN) was an end product of this process, along with wastewater containing 5% nitric acid. Then, the PETN was treated with water, acetone, and ammonium bicarbonate to dissolve and neutralize the free acid present. Mixing equal parts of PETN and TNT produced pentolite. This step also involved the addition of acetone and water. The stoichiometric relationship of the pentolite manufacturing process is as follows:

- $PE + HNO_3 + H_2O \rightarrow PETN + \text{wastewater (nitric acid as 5\%)}$
- $PETN + \text{acetone} + \text{ammonia bicarbonate} + H_2O \rightarrow \text{preliminary filtered neutralized PETN} + \text{wastewater}$
- $(TNT + \text{acetone}) + (PETN + H_2O) \rightarrow \text{pentolite} + \text{wastewater}$

The wastewater from the pentolite manufacturing process potentially contained explosives, metals and acetone. Wastewater from each stage of the manufacturing process was conveyed to two settling basins or the PAWL via vitreous sewer pipes. A wastewater ditch (15 inch channel pipe) encircled the entire perimeter of the lagoons. Wastewater from the first two pentolite lines, along with wastewater from the acetone recovery house, entered the lagoon at the west inlet to the wastewater ditch. Wastewater from the third pentolite line entered the lagoon at the east inlet to the wastewater ditch. Once inside the ditch, the flow of the wastewater was north to south. Three inverts, located at the north and south ends of the lagoons, fed the wastewater into two timber rising wells filled with limestone. These limestone beds were apparently filters and extended 35 ft into the lagoons. It appears that the remainder of the basins further settled the wastewater until it eventually passed over the outlet weir and was pumped to a secondary treatment

facility. Specific wastewater details, treatment processes, and sludge disposal methods are not known.

The PAWL were constructed of pre-cast concrete blocks (15 ft. by 9 ft. by 4 in. thick) with asphalt expansion joints, and each lagoon measured approximately 112 ft. by 137 ft. Four to six inches of No. 4 gravel had underlain the PAWL.

Based on the Site Inspection Report (Morrison Knudsen, 1994), the Pentolite Area was decommissioned in 1945 when ordnance manufacturing ended, and all of the buildings in the area were supposed to be removed or burned in place according to the decontamination procedures. Decontamination of pentolite manufacturing lines was halted during the last quarter of 1945; an estimated 65 % of the necessary decontamination of PBOW was completed by December 1945. The "Shut Down and Decontamination Procedures for PBOW, Sandusky, OH," (Dykema and Lee 1944) states that the stand by and storage procedure for the pentolite settling basin would have consisted of the following:

1. Drain and flush limestone bed and basin proper with a high-pressure hose.
2. Remove limestone while it is completely wet and flush basin thoroughly after removal.
3. Inspect basin thoroughly for evidence of accumulated explosives.

Based on the Records Reviews Report (Dames & Moore, 1997), there is no information regarding decontamination of surrounding soils in the Pentolite Area. Furthermore, the decontamination procedures do not mention the removal of the concrete slabs of the PAWL. An aerial photograph from 1956 shows that the lagoons were still intact. However, this aerial photo indicates that the vitreous sewer pipes leading to the lagoons were removed. From the decontamination procedures, it is assumed that these lines were flushed with water and then cleaned with acetone.

According to SAIC, the PAWL remained essentially intact until 5 July 1956 when approximately 500 acres (including the Pentolite Area and PAWL) was leased by the NASA from the Department of the Army to construct and operate the Plum Brook Reactor Facility. The reactor was planned to be a scientific investigation reactor where the effects of radiation on various materials could be measured. The entire Pentolite Area was filled and graded to facilitate construction of the PBRF. According to Dames and Moore, a memo by Everett and Campbell in 1958 states that the Pentolite Area of approximately 117.3 acres was decontaminated, demolished, and cleared for use as the Lewis Laboratory Reactor Facility. A 1959 historic topographic quadrangle reveals the PBRF with no evidence of the PAWL.

No previous investigations have been performed at the waste lagoons. However, an Underground Storage Tank Corrective Actions Remedial Investigation, Feasibility Study, Phase I Report (Ebasco Environmental, 1991) was performed in

the vicinity of the lagoons. Six monitoring wells were installed, and groundwater and soil samples were collected. Four VOCs were detected in the soil samples.

1.3 Location

The PBOW is located near Sandusky within Erie County, Ohio (Figure 1). The waste lagoons were located within the northern portion of PBOW, specifically, within the Pentolite Area, north of Pentolite Road and south of the present Plum Brook Reactor Facility (PBRF). The Layout of the PAWL is given on the Boring Location Plan (Figure 2).

1.4 Climate

The climate for Erie County is continental with cold and cloudy winters and warm and humid summers. The county's first and last freezing temperatures are typically in October and April, respectively. Average annual precipitation for Sandusky from 1961 to 1990 was 34.05 inches. Within that time period February had the lowest mean monthly rainfall average with 1.65 inches, whereas July had a high of 3.70 inches. The weather changes every few days as cold fronts move through the region. Wind is from the southwest 55 percent of the time (Morrison Knudsen 1994, Dames & Moore 1997).

1.5 Topography

According to historic plans obtained from the previously referenced PA performed by SAIC in 1991, the surface elevation of the waste lagoons was originally 626.15 feet above mean sea level (msl). The perimeter of the waste lagoon angled at a slope of about 2:1 to a bottom elevation of 620.5 msl. The surface of the area that presently occupies the former PAWL is relatively flat at an elevation of about 630 feet above (msl). Based on review of historic topographic quadrangles (USGS, 1959, 1969 and 1979), the surface elevation in the former PBOW has not changed significantly since 1959.

1.6 Geology

1.6.1 PBOW (Installation wide)

Based on the Site Wide Ground Study (IT Corporation, 1997-1998), three formations, all of the Devonian Age, underlie the former PBOW site. The Delaware Limestone is the lowermost formation. It is characterized as a hard, dense, finely crystalline limestone and dolomite. Dissolution of this unit has been described which has produced solution channels along bedding planes and joints, and even producing caverns in some areas. The unit is typically buff colored and usually described as fossiliferous. In the vicinity of PBOW, benzene, toluene, ethyl benzene, and xylene (BTEX) and hydrogen sulfide are common in area

quarries. Overlying the Delaware Limestone is the Olentangy Formation. Two members of the Olentangy Formation have been characterized at the PBOW site, the Plumbrook Shale and the overlying Prout Limestone. The Plum Brook Shale is interpreted to consist of approximately 35 feet of bluish-gray, soft, fossiliferous shale containing thin layers of dark, hard, fossiliferous limestone. The Prout Limestone has been interpreted to be a unit approximately 15 feet thick which outcrops occasionally in a 1,000 to 2,000 foot-wide, northeast striking band across the middle portion of the PBOW. It has been described as a dark-gray to blue, very hard, silicious, fossiliferous limestone or dolomitic mudstone. The uppermost formation at the PBOW site is the Ohio Shale. Only one member of the Ohio Shale is present in the PBOW area- the Huron Shale. This unit has been described as black, thinly bedded, with pyrite and abundant carbonaceous matter with some large pyrite/carbonate concretions up to 6 feet in diameter.

The bedrock overburden in Erie County is predominantly glacial till, glacial outwash or glacial lacustrine (lake) deposits. In the vicinity of PBOW, the soil has been interpreted to be lacustrine. In many areas, the overburden also consists of highly weathered bedrock. The thickness of the overburden ranges from approximately 5 feet or less for most of PBOW to greater than 25 feet. The overburden is thickest on the northern portion of the site.

1.6.2 PAWL (Site Specific)

A subsurface boring record (Dames and Moore, 1997), located about ¼ mile east of the PAWL site, indicated a subsurface profile consisting of an upper layer of brown fine sand extending from the ground surface to a depth of approximately 6 ft bgs. Based on the review of historical grading plans, this upper layer may be fill material associated with the construction of the PBRF. This fill may be over the PAWL site based on the historic grading plans reviewed. The presence of this fill is further indicated by review of the United States Department of Agriculture (USDA) Soil Conservation Service, Soil Survey of Erie county dated 1971. This upper layer is generally underlain by a layer consisting of silty clay and clayey silt soils extending to limestone bedrock. Shale fragments were encountered in the overburden soils from 14 feet to 24 feet bgs. Limestone bedrock was encountered at a depth of about 24 ft bgs.

1.7 Hydrogeology

1.7.1 PBOW (Installation Wide)

Based on the Site Wide Groundwater Study (IT Corporation 1997-1998), potable groundwater is encountered in the bedrock units underlying the PBOW site. Generally this groundwater flows northward toward Lake Erie. Based on published hydrogeologic information (Groundwater Resources of Erie County, 1986), the PBOW site includes 3 distinct hydrogeologic regimes. Groundwater yields from

these regimes range from limited, to the northeast and south, to more than 500 gallons per minute (gpm), to the northwest.

1.7.2 PAWL (Site Specific)

It is anticipated that groundwater in the overburden soils beneath the PAWL site would be perched or trapped water. Perched water occurs in irregular, discontinuous granular zones within the soil overburden. Perched water sources contain widely varying quantities of water depending on recent precipitation and other site-specific factors. Based on the soil types expected, overburden groundwater quantities should be minimal.

Based on the aforementioned hydrogeologic information, the PAWL site is in a hydrogeologic regime that reports yields of 15 gpm, or less from wells drilled into the limestone. Hydrogen sulfide may be present in varying amounts.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 General

This work is being pursued by the United States Army Corps of Engineers (USACE) as part of the Defense Environmental Restoration Program (DERP)/ Formally Used Defense Sites (FUDS). The primary responsibility for the project lies with the Huntington District (CELRH) that acts as the administrator of the funds and performs the overall management functions. CELRH has tasked the Louisville District (CELRL) to execute the design, fieldwork and technical reporting. Several individuals, listed below will coordinate efforts to carry on the project.

2.2 Team Members

Industrial Hygienist:	Shelton Poole, CELRL-ED-EB
Project Engineer:	Chris Karem, CELRL-ED-EE
Safety QC:	Shirley Dunn, CELRL-SO
Risk Assessor:	David Brancato, CELRL-ED-EE
Hydrogeologist:	Martin Wahking, CELRL-ED-EB
QA Chemist:	Samir Mansy, CELRL-ED-EB
Independent Technical Review:	Doug Meadors, CELRL-ED-EE
Project Manager:	Rick Meadows, CELRH-DL-M

3.0 SCOPE AND OBJECTIVES

The purpose of this limited SI is to evaluate the potential for contamination of the site due to past Army activities. Historical information was utilized to identify environmental media and locations most likely to be affected. Field sampling and chemical laboratory analysis will be performed to evaluate the suspect media. Results of the laboratory analysis will be compared to risk based, media specific screening criteria. USEPA Region 9 Preliminary Remediation Goals (PRGs) will be used for the screening criteria. Comparison to background values is beyond the scope of this project.

The Field investigation for this SI will include:

- A visual survey to assess and document site conditions
- Surface and subsurface soil sampling

Target parameters include VOCs, TAL Metals, and Explosives. These parameters will be identified based on analytical results using United States Environmental Protection Agency (USEPA) SW-846 methodology. This data will be used to evaluate the potential for contamination at the site by comparing results to the aforementioned screening criteria. (Region 9 PRGs).

Additional data will be gathered to help assess the physical characteristics of the site and potential migration characteristics of identified mission related contaminants. This includes pH, total organic carbon (TOC) and soil grain size distribution and/or soil plasticity.

4.0 SAMPLING DESIGN AND RATIONALE

Formal Data Quality Objectives (DQOs) for this SI were evaluated and documented in an associated document dated May 1999. Based on historical operations at the PAWL, the COC that were potentially released to the environment include explosives, acetone, and metals. Therefore, samples will be analyzed for explosives, TAL metals, and VOCs. In addition, Total Organic Carbon (TOC) analysis, pH, and sieve and/or plasticity testing will be performed on selected samples in order to assess the soil characteristics at the site.

Based on historical construction drawings, the elevation at the bottom corners of the waste lagoons was about 620.5 ft msl. The elevation of the top of the lagoons was about 626 ft msl. The ground surface in this area is presently at about 630 ft msl.

The bottom of the waste lagoons consisted of a 4 inch thick concrete pad with 4 to 6 inches of gravel subbase. It is believed that the PAWL were removed during construction of the PBRF; however, there is a potential that the PAWL are still intact and fill material was simply placed over the lagoons during construction of the PBRF. Also, affected soils may have been redistributed to any elevation, including the ground surface, during these grading operations.

Based on the nature of the COC and expected grading operations associated with the decommissioning of the waste lagoons and construction of the PBRF, affected media, if present, would most likely be encountered within or just below the fill placed to achieve present grades.

The site will be sampled by advancing 5 to 7 borings in the vicinity of the former PAWL at the approximate locations indicated on the Boring Location Plan. Borings will be advanced to depths ranging from 10 to 15 feet bgs. Continuous soil samples will be collected with a direct-push hydraulic sampler. Each soil sample obtained will be field screened for VOC and TNT and field classified in accordance with the Unified Soil Classification System. Samples selected for chemical laboratory analysis will be based on field observations and screening results. Sampling procedures are detailed in Section 5.4 herein.

5.0 FIELD ACTIVITIES AND SAMPLING PROCEDURES

5.1 Visual Inspections

A visual inspection of the site will be performed before actual sampling takes place. The inspection will consist of a thorough walkover of the site to familiarize the working team with the site and locate visual signs of contamination and potential migration pathways. Modifications to the sampling design may result from this action.

5.2 Sample Documentation

Logbooks with sequentially numbered pages will be kept at the site during all field activities and will be assigned to each sample team. These logs will be updated continuously. Information to be recorded in the logs includes, but is not limited to the following:

- Project Identification.
- General work activity, work dates, and general time of occurrence.
- Unusual events.

- Communication with facility representative.
- Visitors on site.
- Sample number and time of day for each sample collected for analysis.
- Record of telephone calls informing laboratory of sample shipment.
- Variances from project plans and procedures.
- Accomplishment of tailgate safety meetings.
- Photographs taken (including location, spatial orientation, and a brief description of the photograph subject).

5.3 Photographs

Color photographs will be taken of sampling areas to record significant field observations or to record site conditions in the case of visual inspections. Pictures will be logged in the field logbook to identify each picture taken. Prints will be identified with the project number, date and time taken, and a brief description of the subject, location, and orientation of the photograph.

5.4 Soil Sampling Procedures

The soils will be investigated by advancing approximately five soil borings within the former waste lagoon area. The borings will be advanced using a direct-push hydraulic sampler. Continuous soil samples will be obtained in 2 to 4-foot intervals until the boring termination depth is achieved or refusal is encountered. Boring termination depths will vary from 5 to 15 feet depending on observations at the time of the borings. Based on previous borings in the vicinity, bedrock refusal will be encountered at approximately 25 feet bgs. Approximate boring locations are given on the Boring Location Plan (Figure 2).

Field headspace and TNT screening will be performed on a portion of each sample interval. Headspace screening will be performed using a Photoionization Detector (PID). Field TNT screening will be performed using an EnSys Test Kit. Field screening for radionuclides will also be performed. Due to the former operation of the reactor facility by NASA near the site, field health physics measurements shall be provided from the duration of drilling activities. Field screening descriptions are attached to this document.

Selected subsurface soil samples will be sent, under chain of custody protocol, to an off site laboratory for chemical analysis. Chemical analysis of each laboratory sample will

include VOCs, TAL Metals, and Explosives. Testing for pH, TOCs, and Sieve/plasticity analysis will also be performed for every change of soil strata. Samples selected for chemical analysis will be based on the interval most likely to be contaminated from the organoleptic indications and/or field screening results. Laboratory geotechnical analysis will include Atterberg limits and/or Sieve analysis depending on the physical characteristics of the soil. Selection of geotechnical samples will be determined in the field in order to validate field USCS classifications.

5.5 Surface Water and Sediment Sampling

No surface water or sediment samples will be collected for this SI.

5.6 Field Quality Control Sampling

The following field quality control samples will be collected to monitor sampling precision, cross contamination, and decontamination procedures:

- Duplicates - Duplicate samples will be collected at the same time as the original sample and in the same analytical sequence. One field duplicate will be collected for every 10 primary samples. Duplicate samples will be used to monitor sampling precision in the field. Duplicate analysis will be performed for VOC, Metal, and Explosive analyses.
- Rinsate - Rinsate samples will be collected by rinsing sampling equipment with deionized water after decontamination has been performed. The water being used to rinse the equipment will be collected in the appropriate sample container. One rinsate will be collected for every 20 investigative samples. These samples will monitor field-sampling procedures for decontamination completeness. Metal analyses will be performed on rinsates.
- Temperature Blanks - These samples will be prepared by submitting a bottle prefilled by the analytical laboratory. Coordination has been accomplished with the receiving laboratory to ensure that adequate temperature blanks will be provided to the field samplers. These samples will be clearly identified as a temperature blank. This sample will be added in every cooler prepared for shipment to the analytical laboratory to monitor temperature of the samples while in transit from the field to the laboratory.

5.7 Decontamination

Decontamination procedures are implemented to prevent cross contamination, to control potential migration of chemical constituents, and to prevent worker exposure to chemicals or pathogens that may contaminate clothing or protective gear. A decontamination system will be established to wash and rinse all personal protective and sampling equipment. Several gallons of clean, distilled water will be maintained on site along with plastic buckets, brushes, soap, etc., to decontaminate during the sample collection process.

Personal safety and health considerations are presented in the Site-specific Health and Safety plan that was submitted along with this Field Sampling Plan. In addition, all hand tools and equipment will require decontamination prior to removal from the work area. Any materials generated during the site investigation activities (i.e. investigative derived wastes) as a result of decontamination procedures will be labeled and stored until final disposal arrangements, consistent with applicable environmental requirements, are made (see Section 7.0 IDW).

Only minor decontamination of site personnel is recommended, incorporating gross decontamination of the soles of work boots and any personal protective equipment used while on site. All discarded materials shall be handled in such a manner as to preclude spreading of contamination, creating a sanitary hazard, or littering the site. In addition, site workers must wash their hands (and face optional, if exposure warrants) with soap and water before eating, drinking, and before leaving the investigative area.

Decontamination procedures involved in this site investigation will generally involve the subsequent cleaning of any sampling equipment associated with soil collection. Generally accepted measures for ensured data quality and reliability will be employed, specifically involving rinsing of sampling tools and equipment with distilled water and soap (Alconox or other non-phosphate detergent), with a final rinse of deionized water.

This will be accomplished by moving the equipment to a "contained area" and washing down all suspected equipment with brush scrubbing and the soap solution. Hand tools, trowels, scoops, bowls, bailers, etc. used for sample collection of soils shall similarly be decontaminated between samples and before leaving the site for the day.

Decontamination of the Direct Push Rig will first require personnel to dress in suitable safety equipment to reduce personal exposure as required by the Site-Specific Safety and Health Plan.

The Rig itself will require decontamination on various levels. The first level of decontamination is removal of drill cuttings and/or caked on soil. This will be scraped with a flat-bladed scraper at the sampling site. The second level of decontamination is spraying the rig with an Alconox solution and vigorously brushing the area to be cleaned.

Following decontamination, drilling equipment will be placed back on the clean drill rig. If the equipment is not used immediately, it should be stored in a designated clean area.

Rinsates and decontamination fluids will require containerization in containers approved for liquids, labeled and properly stored, while awaiting approval for disposal. Based on the anticipated levels of contamination on most sites, it is believed that disposal approvals will permit disposal of decontamination fluids through the local sanitary sewer.

Materials used for decontamination will be compatible and safe for the purpose intended and for site workers. Consistent with the Hazardous Communication Standard, 29 CFR 1910.1200, any chemical materials brought on site will be accompanied by a Materials Safety Data Sheet (MSDS) and kept with the field team.

6.0 FIELD SAMPLE IDENTIFICATION AND CUSTODY PROCEDURES

Proper sample collection and analysis requires the maintenance of chain-of-custody (CoC) procedures. CoC procedures include tracking and documentation during sample collection, shipment, and laboratory processing. A sample is considered to be in an individual's custody if it is:

- In the physical possession or view of the individual party.
- Secured to prevent tampering.
- Placed in a restricted area by the responsible party.

The sampling team leader is responsible for the custody of the collected samples in the field until they are properly packaged, documented, and released to the courier for shipment to the laboratory. The laboratory is responsible for sample custody thereafter. Custody will be documented by using the CoC record initiated for each day that samples are collected. This record will accompany the samples from the site to the laboratory and will be returned to key project personnel with the final analytical report. All personnel with sample custody responsibilities are required to sign, date, and note the time on the CoC record when relinquishing and receiving samples from their immediate custody. Any discrepancies will be noted at this time. All samples will be shipped via overnight courier to the analytical laboratory. Bills of lading will be used as custody documentation during this time and will be retained as part of the permanent sample custody documentation. Sample documentation and custody for field and laboratory activities are detailed in the following sections.

6.1 Sample Containers, Preservation and Holding Times

The laboratory will supply sample containers. Containers will be selected to ensure compatibility with the sample matrix, chemical constituents to be analyzed, and to minimize breakage during transportation. Sample bottle size required, preservatives, and holding times are listed in the table on the following page. Sample containers, blank labels, preservatives, and packing materials will be supplied by the laboratory. Sample labels will be attached to containers and filled out at the time of sampling. The following information will be recorded on each label:

- sample identification number
- collectors initials
- date and time of collection
- preservatives added
- sample type
- depth

Summary Table of Sample Containers, Preservation Methods, and Holding Times for Soils Samples

Parameter	Analytical Method	Quantity	Type	Preservation Methods	Holding Times
VOC's	8260B	5-15	4-ounce, wide-mouth, amber glass, Teflon®-lined cap,	Cool, 4°C	Analysis: 14 days
TAL Metals	(6000/7000) series	5-15	4-ounce, wide-mouth, amber glass, Teflon®-lined cap,	None	6 months mercury - 28 days
Explosives	8330	5-15	4-ounce, wide-mouth, amber glass, Teflon®-lined cap,	Cool, 4°C	Extraction 7 Days Analysis 40 Days
TOC	9060	5-15	4-ounce, wide-mouth, amber glass, Teflon®-lined cap,	Cool, 4°C	Analysis 28 Days
pH	9045C	5-15		None	Analysis 1 day
Sieve Analysis	ASTM D 421/ASTM D 2217	Varies*	16-ounce, wide-mouth, glass	None	None
Atterberg Limits	*ASTM D 4318	Varies*	4-ounce, wide-mouth, glass	None	None

* Quantity is dependent on the soil types and number of soil types encountered during the field investigation.

6.2 Sample Identification

Each sample for analysis will be assigned a unique identification number.

6.3 Sample Packaging

The following procedures will be performed during sample packaging:

- Number of samples will be verified with field logbook documentation.
- Sample labels will be checked for accuracy and legibility.
- All samples will be wrapped in bubble pack, and placed in a sealed zip-locked bag.
- All coolers will have a temp blank so that the temperature can be monitored.
- Samples will be packaged in a thermally insulated, rigid cooler.
- Packing material will be placed in the coolers to prevent breakage.
- Ice will be placed in the cooler for samples requiring $4^{\circ}\text{C} \pm 2^{\circ}$ preservation.
- Each cooler will have its own Chain of Custody (CoC) form reflecting the samples inside.
- The CoC form will be placed in a sealed zip-lock bag, and taped to the inside lid of the cooler.
- The cooler will be closed and sealed with duct tape around both ends, and around the lid.
- Custody seals will be placed in two separate locations on the cooler across the lid and main body of the cooler and signed by the field team leader.

- An addressed courier bill will be placed on the cooler so that shipment of the cooler can take place.

6.4 Custody Transfer and Shipment Procedures

All samples will be accompanied by a CoC form. When the possession of samples is transferred, the individual relinquishing the samples and the individual receiving the samples will sign, date, and note the time of transfer on the CoC document. This record will represent the official documentation for all transfers of sample custody until samples arrive at Quanterra Laboratories, North Canton, Ohio. Samples will be shipped for overnight service by the courier. This will allow for the least amount of time from sampling and analysis, and will ensure that all holding times are met. Notification of sample shipment to the laboratory will be performed by the Field team leader.

Quanterra Laboratory	Phone (330) 497-9396
4101 Shuffel Drive NW	Fax (330) 497-0772
North Canton, OH 44720	

7.0 DISPOSITION OF FIELD INVESTIGATION DERIVED WASTE (IDW)

Investigation derived waste will be minimal for this field activity. All personal protective equipment (PPE) (e.g., Tyvek[®], nitrile or latex gloves) will be placed in a plastic garbage bag and taken to a dumpster for disposal. All decontamination water will be collected and stored in an appropriate storage container, then emptied into a nearby sewer at the end of the field day. Care will be exercised to insure that the sewer is a collection sewer for the local wastewater treatment plant. Written permission has been granted by the City of Sandusky, Department of Utility Services. The volume of rinsate water is expected to be limited to amounts that can be moved by hand. IDW will be identified and properly handled while it is being accumulated or stored on site.

IDW shall be contained and handled in compliance with the following requirements:

Turnings from hand auger work will be neatly abandoned at the boring locations where sampling occurs.

Soil from direct push sampling will be neatly abandoned at the boring where sampling occurs.

Waste generation will be minimized whenever possible and feasible.

Voids created in the upper portion of holes from direct push activity will be filled with granular bentonite.

Any IDW generated that does not meet the above mentioned criteria will be stored at the site of generation or consolidated at a central storage location supplied by NASA-PBS, pending analytical results.

Stored IDW, pending analytical results, will be characterized for appropriate disposal at a licensed disposal facility within 45 days of initiating field activities.

8.0 SCHEDULE

8.1 Start Date

The earliest date for which field activities will begin is 7 June 1999

8.2 Pre-mobilization

The following activities will be completed before field activities begin:

- Site Access - Access has been obtained from NASA by the U.S Army Corps of Engineers to enter Plum Brook Station.
- Security - Access to Plum Brook Station is controlled by the main gate and security office located on Taylor Road. The security procedures for gaining access are vehicle and personnel registration. The Security office will issue vehicle and personnel badges. Only U.S. citizens with picture I.D. can obtain access to the station. All personnel allowed access to Plum Brook Station are required to view a short 10-minute safety and informational video. The security office will also dispense hand radios to all personnel performing fieldwork for safety as well as security reasons.
- Staging and Support Area - NASA has provided the USACE with a staging area within NASA's shipping and receiving building, Building 9209, located south of Maintenance Road in the garage /maintenance area. This area will be used as a staging area for small sampling supplies and sample shipment.
- Site Visit - A site visit will take place prior to any sampling event. This site visit will allow USACE personnel to visually assess sites, and will facilitate optimization of the sampling design and rationale.

8.3 Mobilization

Mobilization includes efforts required by USACE personnel to prepare for the sampling portion of the site investigation. All sampling team members will review the Field Sampling Plan and Quality Assurance Project Plan prepared for the site investigation. All sampling equipment and materials will be inspected for proper decontamination and good working condition. All provisions will be made by USACE to ensure that field supplies are available and appropriate for sampling team members. These supplies include logbooks, sample containers, labels, chain of custody forms, shipping supplies, coolers, and packing materials.

8.4 Demobilization

At the completion of sampling activities, USACE personnel will demobilize. Arrangements have been made for the disposal of investigation-derived waste (IDW). USACE personnel will prepare waste manifest if necessary. All sampling equipment and materials will be removed by USACE personnel from the site as well as the staging area provided by NASA. USACE personnel will maintain a clean and safe work environment at the investigative site as well as the staging areas provided by NASA. All efforts will be made to leave investigative areas in the same condition as they were found.

Attachment 1

Figures

Figure 1: Site Location Plan

Figure 2: Boring Location Plan

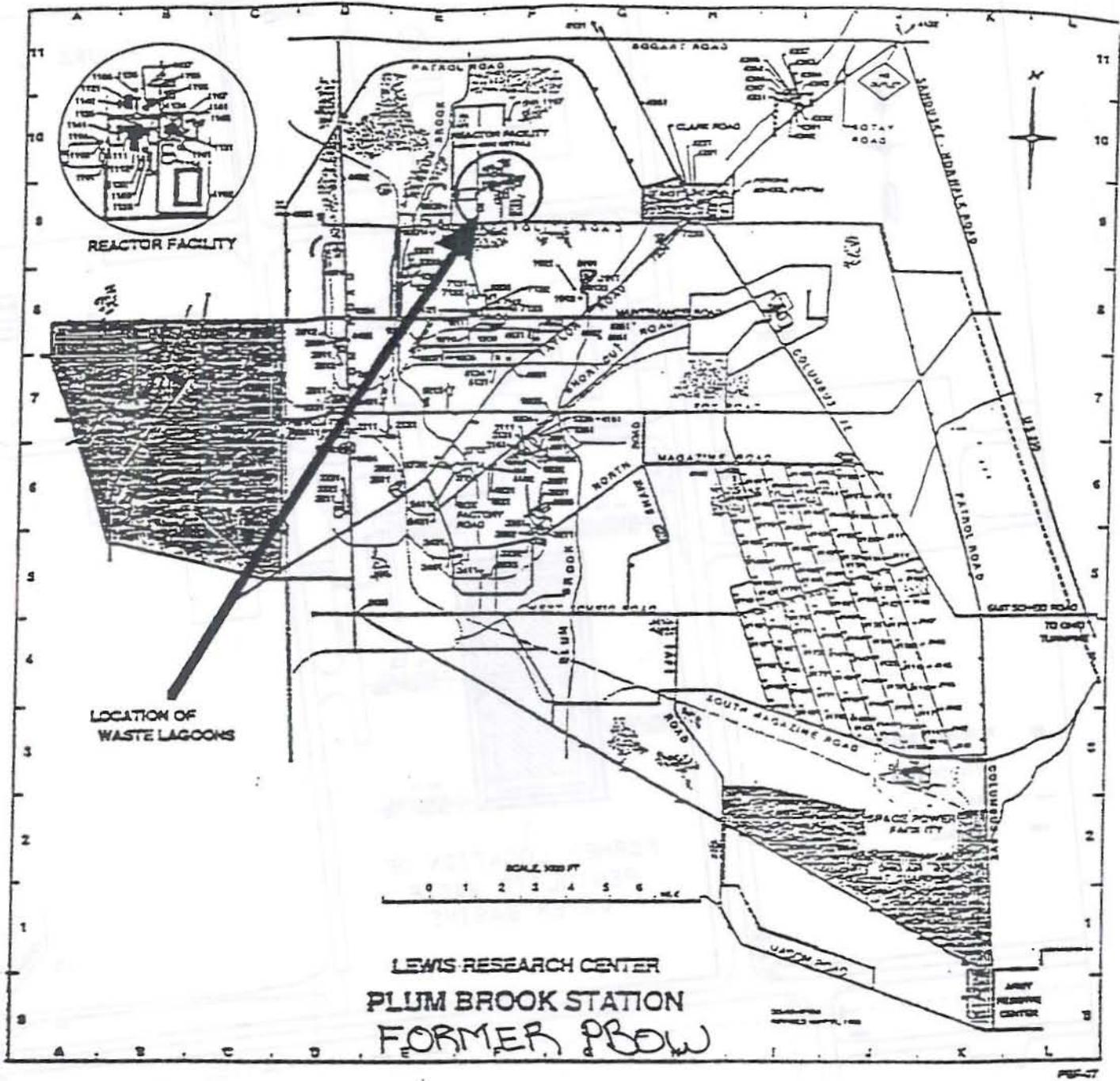
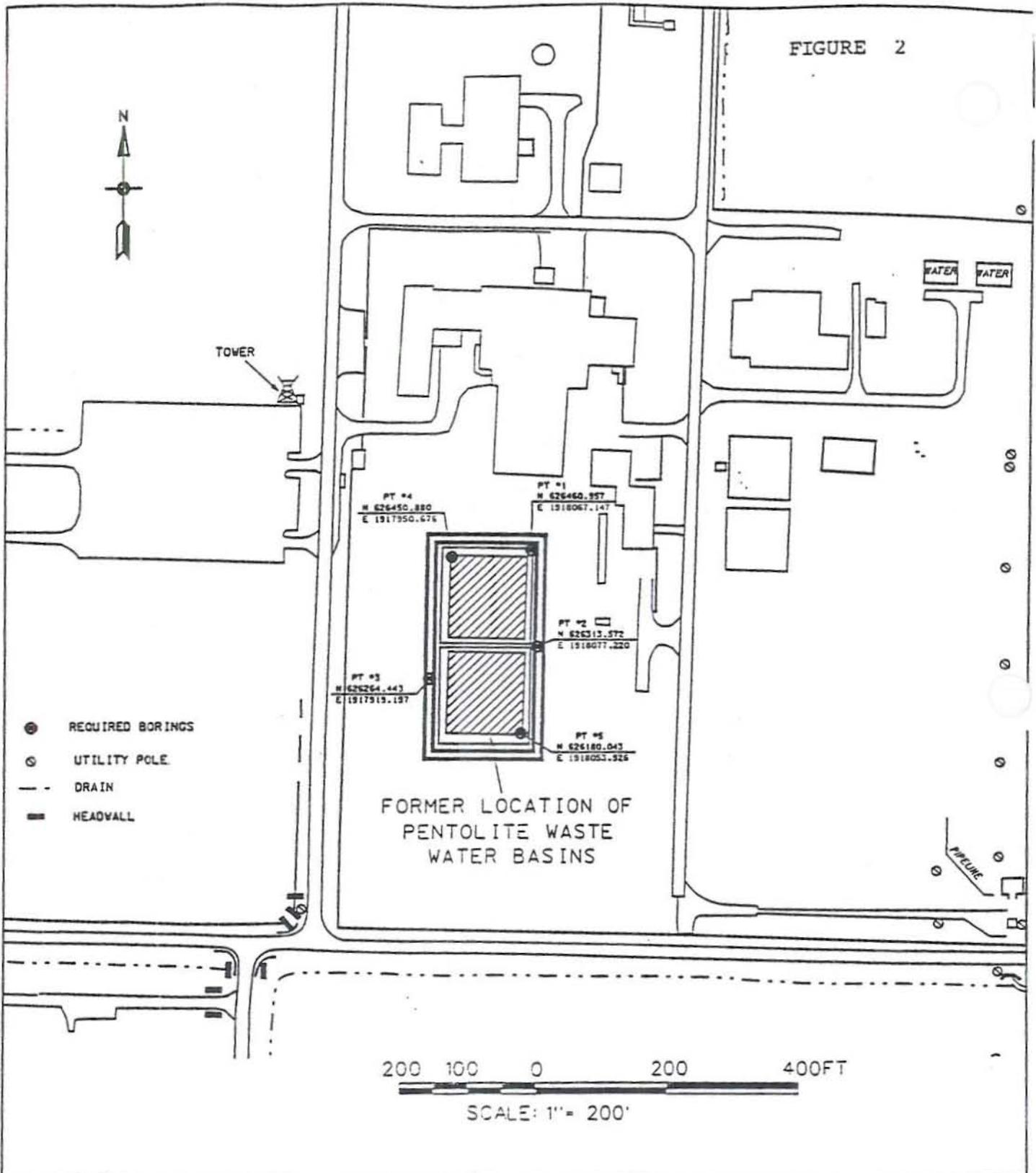


FIGURE 5-11. LOCATION OF WASTE LAGOONS

FIGURE 2



Design By:	Drawn By:	Checked By:	 US Army Corps of Engineers	PLUM BROOK STATION SANDUSKY, OHIO LIMITED SITE INVESTIGATION FY 99	PROPOSED SAMPLE LOCATIONS PENTOLITE AREA WASTE LAGOONS
Reviewed By:	Approved By:				
Date: 10 MAY 99	Scale: 1" = 200'	Drawing Code:	Sheet Ref. No.:	Sht 4 of 7	

File Name: Plot Data

Attachment 2

Field Screening Procedures at Pentolite Area Waste Lagoons

1. Field Headspace Screening
2. Field TNT Screening
3. Field Radionuclide Screening

Field Headspace Screening

2.1 Equipment List

The following list of equipment will be needed to headspace analysis of soil samples:

- clean glass sample containers
- Paper towels
- Aluminum foil
- Organic vapor analyzer equipped with a photoionization detector (PID)
- Field book
- Waterproof and permanent marking pens

2.2 Field Screening Procedures

A portion of each soil sample collected for headspace analysis will be placed in the appropriate glass container. The container should be filled approximately one-half full. The mouth of the container will be covered with aluminum foil, tightly capped, and the sample matrix will be allowed to equilibrate with the headspace for 15 minutes. Care must be taken in the selection of soils with respect to consistency and sample placement in the container in order to achieve comparability and consistency. All headspace material will be handled as IDW.

The sample headspace in the container shall be analyzed with a photoionization analyzer by removing the lid and inserting the instrument probe through the foil liner. Care must be taken in the selection of appropriate foil, placement of the foil on the container, and removal of the lid so as not to compromise the integrity of the seal. If the seal has been compromised, this will be recorded appropriately or a new sample taken if possible.

2.3 Photoionization Detector (PID)

The selection of a PID as the appropriate organic vapor analyzer shall be based on contaminants of concern and/or ambient conditions at the respective site. It is anticipated that a PID detector will be used for all the work.

2.4 Calibration

The instrument(s) selected for use in accordance with data quality objectives and site requirements shall be calibrated according to the manufacturer recommendations and specifications.

2.5 Documentation

All procedures and field conditions will be recorded on the boring log in the field logbook. The record shall include a description of the material being screened and site conditions such as equilibration time and temperature.

Field TNT Screening

Field TNT screening will be performed using an EnSys Test Kit in accordance with the procedures outlined in the following users manual. Field screening for TNT is necessary to insure that a soil with significant explosive contamination is not sent to the laboratory without notice of the significant potential for explosive contamination. Moreover field screening is necessary for the personnel safety of the investigators. TNT is capable of detonating from pressure. The edges of a drill rig or a direct push probe can exert high pressures as the tool is advanced through the soil such that detonation is a possibility. The field screening procedure is also addressed the Site Specific Health and Safety Plan (SSHSP) for this project. The field screening uses an immunoassay procedure. The protocol for the procedure suggests that approximately 25 minutes is necessary to perform the test.



STRATEGIC DIAGNOSTICS INC.

TNT EnSys[®] SOIL TEST SYSTEM

RAPID FIELD SCREEN

User's Guide

IMPORTANT NOTICE

The range of this test is between 1 and 30 ppm TNT/TNB/DNT. The relative standard deviation is 8%. The least detectable concentration is 0.7 ppm (TNT).

This test system should be used only under the supervision of a technically qualified individual who is capable of understanding any potential health and environmental risks of this product as identified in the product literature. The components must only be used for the analysis of soil samples for the presence of TNT. After use, the kits must be disposed of in accordance with applicable federal and local regulations.

PHASE 1 TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

ITEMS INCLUDED IN TEST KIT

- | | | |
|--|--|---|
| <input type="checkbox"/> 2 Orange safety goggles | <input type="checkbox"/> 25 Extraction bags | <input type="checkbox"/> 1 TNT control sample |
| <input type="checkbox"/> 1 Assay vial | <input type="checkbox"/> 1 250 ml beaker | <input type="checkbox"/> 25 - 250 ml vials |
| <input type="checkbox"/> 25 250 ml vials | <input type="checkbox"/> 1 Data entry notebook | <input type="checkbox"/> 25 Wash beaker |
| <input type="checkbox"/> 25 Wash beaker | <input type="checkbox"/> 1 - 250 ml graduated cylinder | |

ITEMS NOT INCLUDED IN TEST KIT

- | | | |
|--|--|---|
| <input type="checkbox"/> 2 Washed HACH cuvettes | <input type="checkbox"/> Assay | <input type="checkbox"/> Wash container |
| <input type="checkbox"/> Paper towels | <input type="checkbox"/> Hach DR/2000 or DR/2010 | <input type="checkbox"/> Balance |
| <input type="checkbox"/> 250 ml graduated cylinder | <input type="checkbox"/> Calculator | |

READ BEFORE PROCEEDING

- For some matrices, air drying the soil samples may result in better TNT recovery or more reproducible data.
- A slightly modified protocol should be used if the primary analyte of concern is DNT. Please refer to the modification outlined on page 6.
- It is recommended that a control be run each day. See page 8 for instructions.
- SDI's EnSys® TNT Soil Test System is designed for use with either of Hach models DR/2000 or the newer DR/2010 spectrophotometers. Protocols for use of both instruments are provided in this User's Guide. Ensure the instrument protocol followed is appropriate for the instrument being used.
- The Hach DR/2000 is designed to turn off after a few minutes of inactivity. Press the "READ/ENTER" key every few minutes to prevent DR/2000 from turning off. If DR/2000 turns off, use Reference cuvette to rezero. Newer DR/2000 models and the DR/2010 have an override "constant on" feature that allows the machine to run indefinitely. Refer to the Instrument Operation: Spectrophotometer Setup section of the HACH DR/2000 or DR/2010 User's manuals.

If you are using the TNT test in conjunction with the RDX test it is important to save your sample extracts. They will be used in the RDX test. Remember to cap the extracts tightly after use. An RDX kit without extraction set-ups can be purchased specifically for this purpose.

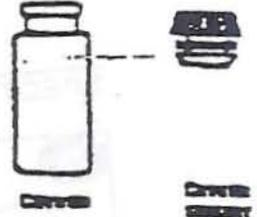
PHASE 1 TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

CLEAN CUVETTES



- 1a Fill 2 Hach matched cuvettes with approximately 5 mL water.
- 1b Cap each with cuvette stopper plug and, holding plug in place, shake vigorously for 3 seconds.
- 1c Empty into waste container.
- 1d Fill cuvettes with approximately 5 mL acetone.
- 1e Cap each with cuvette stopper plug and, holding plug in place, shake vigorously for 3 seconds.
- 1f Empty into waste container.
- 1g Repeat acetone wash (steps 1d - 1f).
- 1h Wipe outside of cuvette with paper towels. Take care to especially clean the side labeled "25 mL" and the side opposite.



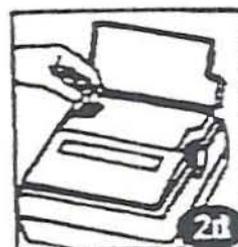
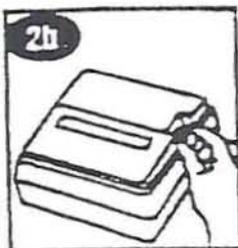
PHASE 1 TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

READ BEFORE PROCEEDING

- Designate a "Reference" and "Sample" cuvette.

SPECTROPHOTOMETER PREPARATION



2a1 Turn on Hach DR/2000. The instrument will read "SELF-TEST" followed by "Method?". Select Method "0" and press the "READ/ENTER" key.

OR

2a2 Turn on the Hach DR/2010. The instrument will read "Self-Test V.xx", then "Enter Program #". Press the [Shift] key (do not hold) and then the [ABS/8] key. Note: Select Program # "0" may also be used to select absorbance mode on the DR/2010.

2b Rotate the wavelength dial until the small display shows: 540 nm.

2c Fill both cuvettes with acetone to the 25 mL line.

2d Insert "Reference" cuvette into cell holder on Hach DR/2000 or DR/2010 with side marked "25 mL" on the right.

2e1 Close light shield of the DR/2000 and press "CLEAR/ZERO" key to establish the reference. The display will read "WAIT" and then "0.000 Abs."

OR

2e2 Close the light shield of the DR/2010 and press the [ZERO] key. The display will read "Zeroing..." then "0.000 Abs."

2f Remove the "Reference" cuvette and place the "Sample" cuvette in the cell holder.

2g1 On the DR/2000, press the "READ/ENTER" key and record the absorbance on the worksheet as "Abs_{background}".

OR

2g2 On the DR/2010, press the [READ] key and record the absorbance on the worksheet as "Abs_{background}".

2h If reading is greater than 0.002 in magnitude (+ or -), clean cuvettes and redo steps 2a - 2g.

2i Empty acetone from "Sample" cuvette into waste container.



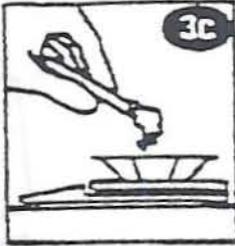
PHASE 2 SAMPLE EXTRACTION & PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

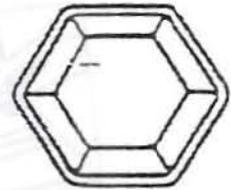
READ BEFORE PROCEEDING

- Sample should be mixed to ensure a homogeneous sample.

WEIGH SAMPLE



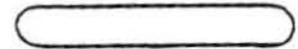
- 3a Place an unused weigh boat on pan balance.
- 3b Press ON/MEMORY button on pan balance. Balance will beep and display 0.0.
- 3c Weigh out 10 +/- 0.1 grams of soil.
- 3d If balance turns off prior to completing weighing, use empty weigh boat to retare, then continue.



Weigh Boat



Pan Balance



Wooden Spatula

EXTRACT TNT



- 4a Measure 50 mL acetone in the 50mL graduated conical tube.
 - 4b Pour acetone into an extraction jar.
 - 4c Using wooden spatula, transfer 10 grams of soil from weigh boat into extraction jar.
 - 4d Recap extraction jar tightly and shake vigorously for three minutes.
 - 4e Allow to settle for five minutes.
- Repeat steps 3a - 4e for each sample to be tested.

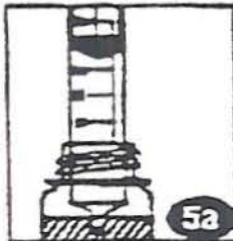


50 mL Graduated Conical Tube



Extraction Jar

FILTER SAMPLE



- 5a Place tip of 30 cc syringe into liquid above the sediment layer in the extraction jar and draw up 25 mL of the sample.
- 5b Screw the syringe filter onto the end of the syringe.
- 5c Press the plunger firmly and dispense the sample into the "Sample" cuvette.



30 cc Syringe



Syringe Filter

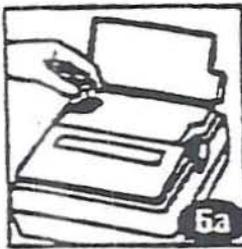


Sample Cuvette

PHASE 3 SAMPLE ANALYSIS

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

READ SAMPLE



- 6a Place the "Sample" cuvette in the cell holder.
- 6b Press the "READ/ENTER" key and record the absorbance on the worksheet as "Abs_{initial}".
- 6c Remove the "Sample" cuvette from the cell holder.
- 6d Add 1 drop of Developer Solution.
- 6e Cap the "Sample" cuvette and shake vigorously for 3 seconds.



DNT Analysis Note:

For analysis of samples containing DNT, and/or where DNT concentration is of concern, samples must be allowed to develop for 10 minutes before reading sample absorbance. This will not effect color development for other nitroaromatics.

- 6f Remove the cuvette stopper and place the "Sample" cuvette in the cell holder.
- 6g Press the "READ/ENTER" key and record the absorbance on the worksheet as "Abs_{sample}".
- 6h Clean cuvette between samples using procedure in steps 1a - 1h.

PHASE 4 INTERPRETATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

INTERPRETATION OF RESULTS

- 7a Multiply the "Abs_{initial}" value for each sample by 4. Enter these values on the worksheet.
- 7b Subtract this value from the "Abs_{sample}" values for each sample and record on the worksheet.
- 7c Divide the adjusted sample value by 0.0323 and record on the worksheet. This value is the TNT concentration of the sample in parts per million.

$$\text{TNT (ppm)} = \frac{\text{Abs}_{\text{sample}} - (\text{Abs}_{\text{initial}} \times 4)}{0.0323}$$

Note: For sample concentrations greater than 30ppm the sample extract should be diluted with acetone and reanalyzed.

Remember to multiply the result by the dilution factor in order to determine the correct concentration.

CONTROL (QA/QC) CHECK

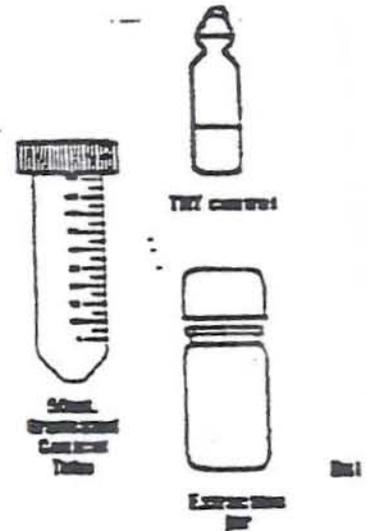
READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

- The TNT control is optional, but it is recommended that it be run daily.

PREPARE CONTROL



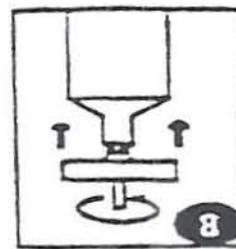
- 1 Measure 50 mL acetone in the 50mL graduated conical tube.
- 2 Pour into extraction jar.
- 3 Open TNT control ampule by slipping ampule cracker over top, and then breaking tip at scored neck.
- 4 Transfer entire contents of TNT control ampule into extraction jar using bulb pipette.
- 5 Cap extraction jar and shake vigorously for 3 seconds.



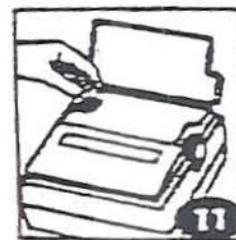
ANALYZE THE CONTROL



- 7 Place tip of 30 cc syringe in extraction jar and draw up 25 mL.
- 8 Attach syringe filter and dispense into "Sample" cuvette.
- 9 Add 1 drop of developer solution.
- 10 Cap the cuvette and shake vigorously for 3 seconds.
- 11 Remove the cuvette stopper and place in the cell holder.
- 12 Press "READ/ENTER" key and record the absorbance on the worksheet as "Abs_{control}".



Absorbance must be between 0.307 - 0.373 for the test to be in control. If test is not in control, clean "Sample" cuvette, and then redo steps 7-12 using the remaining liquid from the extraction jar.



- 13 If test is in control clean "Sample" cuvette before proceeding with samples.



QUALITY CONTROL

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

System Description

Each SDI EnSys® TNT Soil Test System contains enough material to perform twenty complete tests. The TNT Soil Test is divided into four phases. The instructions and notes should be reviewed before proceeding with the test.

Hotline Assistance

If you need assistance or are missing necessary Test System materials, call toll free: 1-800-544-8881.

Validation Information

Product claims are based on validation studies carried out under controlled conditions. Data has been collected in accordance with valid statistical methods and the product has undergone quality control tests of each manufactured lot.

Strategic Diagnostics Inc. does not guarantee that the results with the TNT Soil Test System will always agree with instrument-based analytical laboratory methods. All analytical methods, both field and laboratory, need to be subject to the appropriate quality control procedures.

How It Works

Controls, Samples, and color-change reagents are added to cuvettes. The concentration of TNT in an unknown Sample is determined by evaluating how much color is developed.

Quality Control

Standard precautions for maintaining quality control:

- Do not use reagents or components from one Test System with reagents or components from another Test System.
- Do not use the Test System after its expiration date.
- The sample must be analyzed immediately after adding the Developer Solution.
- Results may not be valid if DR/2000 reading for Control is outside of the range of 0.307 - 0.373.

Storage and Handling Precautions

- Wear protective gloves and eye wear.
- Store kit at room temperature and out of direct sunlight (less than 80°F).
- If acetone comes into contact with eyes, wash thoroughly with cold water and seek immediate medical attention.
- Operate test at temperatures greater than 4° C/40° F and less than 39° C/100° F.
- After use, dispose of kit components in accordance with applicable federal and local regulations.

ON-SITE QUALITY CONTROL/QUALITY ASSURANCE RECOMMENDATIONS SDI EnSys® TEST SYSTEM

Please read the following before proceeding with field testing.

SAMPLING

The result of your screening test is only as valid as the sample that was analyzed. Samples should be homogenized thoroughly to ensure that the 10 grams you remove for field testing is representative of the sample as a whole. All other applicable sample handling procedures should be followed as well.

PRIOR TO TESTING SAMPLES

Carefully follow the instructions in the User's Guide included with every test kit. This is the key element in obtaining accurate results. In addition, store your unused test kits at room temperature and do not use them past their expiration date (see label on each test kit).

INTERNAL TEST QC

One control is provided with each Kit to provide internal test system quality control. Test runs resulting in a number that falls outside of the specified range should be repeated to ensure valid conclusions.

QA/QC

The validity of field test results can be substantially enhanced by employing a modest, but effective QA/QC plan. SDI recommends that you structure your QA/QC plan with the elements detailed below. These have been developed based on the data quality principles established by the U.S. Environmental Protection Agency.

- A. Sample Documentation
 - 1. Location, depth
 - 2. Time and date of collection and field analysis
- B. Field analysis documentation - provide raw data, calibration, any calculations, and final results of field analysis for all samples screened (including QC samples)
- C. Method calibration - this is an integral part of SDI tests; a TNT control analysis should be performed daily (see the instructions in the User's Guide)
- D. Method blank - field analyze fresh acetone
- E. Site-specific matrix background field analysis - collect and field analyze uncontaminated sample from site matrix to document matrix effect
- F. Duplicate sample field analysis - field analyze duplicate sample to document method repeatability; at least one of every 20 samples should be analyzed in duplicate
- G. Confirmation of field analysis - provide confirmation of the quantitation of the analyte via an EPA-approved method different from the field method on at least 10% of the samples; provide chain of custody and documentation such as gas chromatograms, mass spectra, etc.
- H. Performance evaluation sample field analysis (optional, but strongly recommended) - field analyze performance evaluation sample daily to document method/operator performance
- I. Matrix spike field analysis (optional) - field analyze matrix spike to document matrix effect on analyte measurement

FURTHER QUESTIONS?

SDI's Technical Support personnel are always prepared to discuss your quality needs to help you meet your data quality objectives. Call 1-(800) 544-8881.

TNT SOIL TEST - ABBREVIATED PROCEDURE

1	<ul style="list-style-type: none"> • Clean cuvettes • Zero the spectrophotometer at 540 nm
2	<ul style="list-style-type: none"> • Add 10 g soil and 50 ml acetone to extraction jar • Shake 3 minutes, let settle • Draw up 25 mL extract, filter into cuvette
3	<ul style="list-style-type: none"> • Read Abs_{initial}, record • Add 1 drop developer solution, shake • Read Abs_{sample}, record <p style="text-align: right; margin-right: 50px;">TNT TNB DAB 1 to 30 ppm</p>
4	<ul style="list-style-type: none"> • Multiply Abs_{initial} by 4 • Subtract from Abs_{sample} • Divide by 0.0323 • TNT(ppm) = $\frac{\text{Abs}_{\text{sample}} - (\text{Abs}_{\text{initial}} \times 4)}{0.0323}$

TNT likely COC
no need to quantify, don't need Herb
determine the pres/abs. of TNT

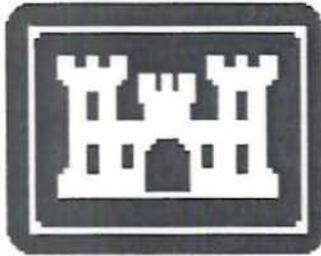
1800
544
8981

ext.
138

Field Radionuclide Screening

Field screening for radionuclides will be performed. Due to the former operation of the reactor facility by NASA near the site, field health physics measurement shall be provided for the duration of the drilling activities. A report completed by SAIC indicates some degree of radiological materials present in drainage ditches in the Pentolite lagoon area. (SAIC, 1998) The publication EM 385-1-1 of 3 Sep 96 provides in Part 06.E.01 procedures for working with radioactive materials. A portable direct reading instrument will be used for monitoring soil samples and labeling samples to alert the analytical laboratory to the potential of radiological material. In addition the health physics activity includes aiding the team in minimizing exposure to the existing ditch area, sensing the body covering of the employees on site and any IDW generated by the operation. Each subsurface soil sample shall be observed as it is recovered, and scanned for radiological material. If radiological material is indicated, further sampling shall be halted and consideration given to alteration of procedures. Screening will insure minimized personnel exposure and proper labeling and interim handling of the containerized samples and any IDW created by the SI activities. The radionuclide hazard is described in the Site Specific Safety and Health Plan.

NASA has an existing program for minimizing radiological contamination from operations in the PBRF area. All tools and materials and personnel brought into the PBRF area must be field screened by NASA before exit of the PBRF area is allowed. This will be the final decontamination check for each event where the field team leaves the PAWL area.



**U.S. ARMY CORPS
OF ENGINEERS
LOUISVILLE DISTRICT**

Data Quality Objectives

Limited Site Investigation of the former

Plum Brook Ordnance Works

**Pentolite Area Waste
Lagoons**

Sandusky, Ohio

DERP-FUDS HTRW Project No. G05OH001819

JULY 2000

1.0 DATA QUALITY OBJECTIVES PROCESS

The purpose of this document is to provide the rationale for developing Data Quality Objectives (DQOs) for the Site Investigation (SI) of the Pentolite Area Waste Lagoons (PAWL) located at the former Plum Brook Ordnance Works (PBOw), Ohio FUDS Site. The former Plum Brook Ordnance Works (PBOw) was operated from 1941 to 1945 by the Trojan Powder Company under contract to the Army Ordnance Department. The facility manufactured trinitrotoluene (TNT), dinitrotoluene (DNT), and pentolite. The PAWL were settling basins for the waste water from the pentolite manufacturing process. Investigation of the PBOw is being addressed by the DOD under the Defense Environmental Restoration Program (DERP), Formerly Used Defense Sites (FUDS) program. This project is being undertaken by the US Army Corps of Engineers Louisville District (CELRL) under the direction of the US Army Corps of Engineers Huntington District (CELRH).

The DQO process is a strategic planning approach based on the Scientific Method that is used to prepare for a data collection activity. It provides a systematic procedure for defining the criteria that a data collection design should satisfy, including when to collect samples, sample collection locations, the tolerable level of decision errors for the study, and how many samples should be collected.

The DQO process consists of the following seven distinct steps (USEPA, 1994):

- Step 1: State the Problem – Concisely describe the problem to be studied. Review prior studies and existing information to gain a sufficient understanding to define the problem.
- Step 2: Identify the Decision – Identify what questions the study will attempt to resolve, and what actions may result.
- Step 3: Identify the Inputs to the Decision – Identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statement.
- Step 4: Define the Study Boundaries – Specify the time periods and spatial area to which decisions will apply. Determine when and where the data should be collected.
- Step 5: Develop a Decision Rule – Define the statistical parameters of interest, specify the action level, and integrate the previous DQO outputs into a single statement that describes the logical basis for choosing among alternative actions.

- Step 6: Specify Tolerable Limits on Decision Error – Define the decision maker's tolerable decision error rates based on a consideration of the consequences of making an incorrect decision.
- Step 7: Optimize the Design – Evaluate information from the previous steps and generate alternative data collection designs. Choose the most resource-effective design that meets all DQOs.

The DQO process is iterative by design; the outputs of one step may influence other steps in the process and improve the investigation as knowledge of the site increases.

2.0 BACKGROUND

2.1 Installation and Site History

Based on the Archives Search Report (USACE, 1993), the original PBS site was established in 1941 and referred to as Plum Brook Ordnance Works (PBOW). The installation was established for the purpose of manufacturing trinitrotoluene (TNT), dinitrotoluene (DNT), pentolite, and nitric and sulfuric acids. Built by E.B. Badger and Sons Company, the facility was operated under contract by the Trojan Powder Company. Production of explosives ceased two weeks after V-J Day, having manufactured in excess of one billion pounds of explosives during the four-year operating period.

By September 1945, the entire Ordnance Inspection Department was abolished. Decontamination of TNT, acid, pentolite and DNT manufacturing lines was completed during the last quarter of 1945. On 17 December 1945, the physical custody of the plant was transferred from Trojan to the Ordnance Department. The U.S. Army Corps of Engineers assumed responsibility for maintenance and custodial duties until September 1946 when the property was transferred to the War Assets Administration (predecessor to the Government Services Administration (GSA)), after it was certified by the U.S. Army to be decontaminated. NASA acquired the PBOW in 1963 and is presently using the site, now referred to as PBS.

The PBS site currently lies in an area that is primarily rural and agricultural with a low population density. The NASA Lewis Research Center occupies a majority of the former ordnance works. The Department of the Army maintains a reserve center on the westernmost portion of the facility. The remainder of the former installation is in private ownership with the vast majority being cultivated. A tract on the northern boundary is owned by the Perkins Board of Education and is utilized as a bus maintenance facility.

The focus of this site investigation will be the former PAWL. Based on the PBS Preliminary Assessment (PA) (Science Applications International Corporation (SAIC), 1991), the first stage of the pentolite manufacturing process involved the nitration of pentaerythritol (PE) by adding nitric acid and water. Pentaerythritol tetranitrate (PETN) was an end product of this process, along with wastewater containing 5% nitric acid. Then, the PETN was treated with water, acetone, and ammonia bicarbonate to dissolve and neutralize the free acid present. Mixing equal parts of PETN and TNT produced pentolite. This step also involved the addition of acetone and water. The stoichiometric relationship of the pentolite manufacturing process is as follows:

- $PE + HNO_3 + H_2O \rightarrow PETN + \text{wastewater (nitric acid as 5\%)}$
- $PETN + \text{acetone} + \text{ammonia bicarbonate} + H_2O \rightarrow \text{preliminary filtered neutralized PETN} + \text{wastewater}$
- $(TNT + \text{acetone}) + (PETN + H_2O) \rightarrow \text{pentolite} + \text{wastewater}$

The wastewater from the pentolite manufacturing process potentially contained explosives, metals and acetone. Wastewater from each stage of the manufacturing process was conveyed to two settling basins referred to as the PAWL via vitreous sewer pipes. A wastewater ditch (15 inch channel pipe) encircled the entire perimeter of the lagoons. Wastewater from the first two pentolite lines, along with wastewater from the acetone recovery house, entered the lagoon at the west inlet to the wastewater ditch. Wastewater from the third pentolite line entered the lagoon at the east inlet to the wastewater ditch. Once inside the ditch, the flow of the wastewater was north to south. Three inverts, located at the north and south ends of the lagoons, fed the wastewater into two timber rising wells filled with limestone. These limestone beds were apparently filters and extended 35 feet into the lagoons. It appears that the remainder of the basins further settled the wastewater until it eventually passed over the outlet weir and was pumped to a secondary treatment facility. Specific wastewater details, treatment processes, and sludge disposal methods are not known.

The PAWL were constructed of pre-cast concrete blocks (15 ft. by 9 ft. by 4 in. thick) with asphalt expansion joints, and each lagoon measured approximately 112 ft. by 137 ft. Four to six inches of No. 4 gravel had underlain the PAWL.

Based on the Site Inspection Report (Morrison Knudsen, 1994), the Pentolite Area was decommissioned in 1945 when ordnance manufacturing ended, and all of the buildings in the area were supposed to be removed or burned in place according to the decontamination procedures. Decontamination of pentolite manufacturing lines was halted during the last quarter of 1945, and it was estimated that 65 % of the necessary decontamination of PBOW was completed by December 1945. The "Shut Down and Decontamination Procedures for PBOW, Sandusky, OH," (Dykema and Lee 1944) states

that the stand by and storage procedure for the pentolite settling basin shall consist of the following:

Drain and flush limestone bed and basin proper with a high-pressure hose.
Remove limestone while it is completely wet and flush basin thoroughly after removal.
Inspect basin thoroughly for evidence of accumulated explosives.

Based on the Records Reviews Report (Dames & Moore, 1997), there is no information regarding decontamination of surrounding soils in the Pentolite Area. Furthermore, the decontamination procedures do not mention the removal of the concrete slabs of the PAWL. An aerial photograph from 1956 shows that the lagoons were still intact. However, this aerial photo indicates that the vitreous sewer pipes leading to the lagoons were removed. From the decontamination procedures, it is assumed that these lines were flushed with water and then thoroughly cleaned with acetone.

According to SAIC, the PAWL remained essentially intact until 5 July 1956 when approximately 500 acres (including the Pentolite Area and PAWL) were leased by the NACA from the Department of the Army. The Plum Brook Reactor Facility (PBRF) was constructed on this site, and consequently, the PAWL were likely removed. The entire Pentolite Area was filled and graded to facilitate construction of the PBRF. According to Dames and Moore, a memo by Everett and Campbell in 1958 states that the "Pentolite Area of approximately 117.3 acres was decontaminated, demolished, and cleared for use as the Lewis Laboratory Reactor Facility". A 1959 historic topographic quadrangle reveals the PBRF with no evidence of the PAWL.

No previous investigations have been performed at the waste lagoons. However, an Underground Storage Tank Corrective Actions Remedial Investigation, Feasibility Study, Phase I Report (Ebasco Environmental, 1991) was performed in the vicinity of the lagoons. Six monitoring wells were installed, and groundwater and soil samples were collected. Four VOCs were detected in the soil samples.

2.2 PAWL Topography

According to historic plans obtained from the previously referenced SAIC report, the surface elevation of the waste lagoons was originally at 626.15 feet above mean sea level (msl). The perimeter of the waste lagoon angled at a slope of about 2:1 to a bottom elevation of 620.5 msl. The surface of the area that presently occupies the former PAWL is relatively flat and at about 630 feet above (msl). Based on review of historic topographic quadrangles (USGS, 1959, 1969 and 1979), the surface elevation in the former PBOW has not changed significantly since 1959.

2.3 Geology

Based on the Site Wide Ground Study (IT Corporation, 1997-1998), three formations, all of the Devonian Age, underlie the former PBOW site. The Delaware Limestone is the lowermost formation. It is characterized as a hard, dense, finely crystalline limestone and dolomite. Dissolution of this unit has been described which has produced solution channels along bedding planes and joints, and even producing caverns in some areas. The unit is typically buff colored and usually described as fossiliferous. In the vicinity of PBOW, benzene, toluene, ethyl benzene, and xylene (BTEX) and hydrogen sulfide are common in area quarries. Overlying the Delaware Limestone is the Olentangy Formation. Two members of the Olentangy Formation have been characterized at the PBOW site, the Plumbrook Shale and the overlying Prout Limestone. The Plum Brook Shale is interpreted to consist of approximately 35 feet of bluish-gray, soft, fossiliferous shale containing thin layers of dark, hard, fossiliferous limestone. The Prout Limestone has been interpreted to be a unit approximately 15 feet thick which outcrops occasionally in a 1,000 to 2,000 foot-wide, northeast striking band across the middle portion of the PBOW. It has been described as a dark-gray to blue, very hard, silicious, fossiliferous limestone or dolomitic mudstone. The uppermost formation at the PBOW site is the Ohio Shale. Only one member of the Ohio Shale is present in the PBOW area- the Huron Shale. This unit has been described as black, thinly bedded, with pyrite and abundant carbonaceous matter with some large pyrite/carbonate concretions up to 6 feet in diameter.

The bedrock overburden in Erie County is predominantly glacial till, glacial outwash or glacial lacustrine (lake) deposits. In the vicinity of PBOW, the soil has been interpreted to be lacustrine. In many areas, the overburden also consists of highly weathered bedrock. The thickness of the overburden ranges from approximately 5 feet or less for most of PBOW to greater than 25 feet. The overburden is thickest on the northern portion of the site.

A subsurface boring record (Dames and Moore, 1997), located about ¼ mile east of the PAWL site, revealed a subsurface profile consisting of an upper layer of brown fine sand extending from the ground surface to a depth of approximately 6 ft bgs. Based on the review of historical grading plans, this upper layer may be fill material associated with the construction of the PBRF. This fill may be over the PAWL site based on the historic grading plans reviewed. The presence of this fill is further indicated by review of the United States Department of Agriculture (USDA) Soil Conservation Service, Soil Survey of Erie county dated 1971. This upper layer is generally underlain by a layer consisting of silty clay and clayey silt soils extending to limestone bedrock. Shale fragments were encountered in the overburden soils from 14 feet to 24 feet bgs. Limestone bedrock was encountered at a depth of about 24 feet bgs.

2.4 Hydrogeology

Based on the Site Wide Groundwater Study (IT Corporation 1997-1998), potable groundwater is encountered in the bedrock units underlying the PBOW site. Generally this groundwater flows northward toward Lake Erie. Based on published hydrogeologic information (Groundwater Resources of Erie County, 1986), the PBOW site includes 3 distinct hydrogeologic regimes. Groundwater yields from these regimes range from limited, to the northeast and south, to more than 500 gallons per minute (gpm), to the northwest.

It is anticipated that groundwater in the overburden soils beneath the PAWL site would be perched or trapped water. Perched water occurs in irregular, discontinuous granular zones within the soil overburden. Perched water sources contain widely varying quantities of water depending on recent precipitation and other site-specific factors. Based on the soil types expected, overburden groundwater quantities should be minimal.

Based on the aforementioned hydrogeologic information, the PAWL site is in a hydrogeologic regime that reports yields of 15 gpm, or less from wells drilled into the limestone. Hydrogen sulfide may be present in varying amounts.

3.0 Data Quality Objectives Process

3.1 Step 1 – State the Problem: The purpose of this step is to define the problem so that the focus of the study will be unambiguous. Concisely describe the problem to be studied and review prior studies and existing information to gain a sufficient understanding to define the problem.

3.1.1 Identification of Planning Team Members:

Project Manager:	Rick Meadows, CELRH-DL-M
Project Engineer:	Chris Karem, CELRL-ED-EB
Industrial Hygienist:	Shelton Poole, CELRL-ED-EB
Safety QC	Shirley Dunn, CELRL-SO
Risk Assessor:	David Brancato, CELRL-ED-EE
QA Chemist:	Samir Mansy, CELRL-ED-EB
Independent Technical Review:	Doug Meadors, CELRL-ED-EE Martin Wahking, CELRL-ED-EB

3.1.2 Description of Problem: Potential contamination at the PAWL was identified in a project summary sheet, as an exhibit to an Inventory Project Report (INPR) prepared by Huntington District, which requested a Limited Site Investigation. The purpose of the Limited SI was to identify any contamination related to the previous Army activities. Soil samples have not been collected in this area.

3.1.3 Potential Transport Mechanisms: Potential transport mechanisms in the PAWL include:

- Surface runoff to Hemming Ditch.
- Leaching through the soil column to the subsurface soil and groundwater.

3.1.4 Resources and Relevant Deadlines for the Site Investigation:

The funding resources for Site Investigations are provided under the DERP-FUDS DOD program. This limited SI must be completed by the end of FY 99 in order to closeout the records for this project in the financial system. The Louisville District Environmental Engineering Branch is slated to provide the necessary labor to complete the investigation. This is slated to be a limited SI since it is the initial investigative work for the PAWL.

3.2 Step 2: Identify the Decision – The purpose of this step is to define the decision statement that the study will attempt to resolve.

3.2.1 Principal Study Question – Do constituents of concern exist in the environmental media at the PAWL at levels that would exceed those found in USEPA Region IX PRGs?

3.2.2 Alternative Actions that could result from Resolution of the Principle Study Question:

- SI report recommendation: Coordinate with federal and state regulatory authorities to proceed toward a no further action (NFA) decision document.
- SI report recommendation: Additional site investigation or interim measures.

3.2.3 Decision Statement – The primary decision for the site investigation is to determine whether the COCs present at the PAWL are at levels that exceed media specific screening criteria (Region IX PRGs) and thus would require further action.

3.3 Step 3: Identify Inputs to the Decision – The purpose of this step is to identify the informational inputs that will be required to resolve the decision statement and determine which inputs require environmental measurements.

3.3.1 Information Required to Resolve Decision Statement:

- Historical records, interviews, aerial photographs, visual inspections and previous environmental investigations will be utilized to make an informed decision about the expected type of COCs. This information in conjunction with site topography, geology, site hydrology and hydrogeology will help determine locations where contaminated media will be discovered.
- Transport mechanisms and chemical properties of COCs to evaluate migration pathways.
- Analytical samples and results from the corresponding environmental media within the PAWL to compare to PRGs.

3.3.2 Sources for Information – DERP-FUDS and EPA guidance are the principle tools leading this investigation. CELRH has an extensive administrative record (AR) for the former PBO. A review of excerpts from this AR and other sources including historic aerial photographs, historical topographic quadrangles, published geologic information and data base searches revealed useful information about the site. Information regarding the chemical properties and characteristics of COCs can be obtained from the NIOSH *Chemical Guide* (NIOSH 1998). The “References” section of the Quality Assurance Project Plan (QAPP) dated June 1999 for this project contains a complete listing of the reviewed information.

3.3.3 Information Needed to Establish the Action Level and Confirm that Appropriate Measurement Methods Exist to Provide the Necessary Data – The action levels for the contaminants of concern (COCs) will be the USEPA Region IX screening criteria for the protection of human health. USEPA SW-846 analytical methods were selected for sample analysis to provide Method Detection Limits that are sufficiently low enough to allow comparison with applicable screening criteria. The performance-based methods have inherited quantitative and qualitative QA objectives, internal method requirements, and specific QC limits. These methods along with strict USEPA QA/QC guidance and protocols will provide data that will meet data quality objectives.

3.4 Step 4 – Define Site Investigation Boundaries – This step describes the spatial and temporal boundaries of the site investigation to which decisions will apply. Characteristics of the population to be sampled are defined, and practical considerations for the site investigation are evaluated in this section. Based on the initial results of the

site investigation, additional data may be required to further define the investigation boundaries.

3.4.1 Characteristics that Define the Population of Interest – The COC associated with the wastewater at the lagoons that may have been released into the environment include explosives, metals and acetone. Because the COC may exist in different soil strata, more than one stratum should be characterized by sampling. Samples should be collected from the surface soil to characterize the population of COC that may have been redistributed by construction activities at the Plum Brook Reactor Facility (PBRF). Samples should also be collected within the location of the former lagoons at and below the elevation of the bottom of the former lagoons to evaluate constituents that may have been left in place or migrated downward through the soil column.

3.4.2 Spatial Boundary of the Decision Statement – The site investigation will focus on the PAWL, identified in historical drawings, aerial photos, and site visits. The approximate size of the PAWL is 1.3 acres. The Horizontal extent of the investigation is shown on the Boring Location Plan (see Field Sampling Plan (FSP)). The vertical extent of contamination is to bedrock, which is anticipated to be about 25 feet bgs.

3.4.3 Temporal Boundary of Decision Statement – The analytical data obtained from this site investigation will be used as valid indicators of COCs throughout an exposure time frame of 50 years. The sampling for this investigation should take place in the time frame of early spring through summer. If performed in this time frame, optimum weather conditions for fieldwork should occur.

3.4.4 Scale of Decision Making – The scale of decision making will be based on the concentrations of the possible contaminants identified in the surface water, surface soil, and subsurface soil samples compared to the values for the Region IX PRGs.

3.4.5 Practical Constraints on Data Collection – Practical constraints on data collection for this site investigation could be scheduling problems, access problems, personal injury during fieldwork, illness, dangerous weather, and/or budget constraints.

3.5 Step 5: Develop a Decision Rule – Define the statistical parameters of interest, specify the action level, and integrate the previous DQO outputs into a single statement that describes the logical basis for choosing among alternative actions.

3.5.1 Specify the Parameter that Characterizes the Population of Interest – The concentration levels of the COCs found will be compared to the levels of the specific screening criteria. Any findings surpassing the criteria levels will become the area of focus for further investigation.

3.5.2 Specify the Action Level for the Site Investigation – The analytical sample results will be compared to the proper screening standard set forth by Region IX PRGs. The levels of screening are conservative so that the proper protection is met for both human health and the environment.

3.5.3 Decision Rule – If concentration levels of the COCs are higher than that of the criteria levels, then a recommendation for further investigation and or remediation will be implemented. If the concentration levels are below the criteria, then an approach for a No Further Action (NOFA) will be developed. During the review process data gaps may be uncovered. Such data gaps may require additional media sampling and analytical chemistry effort to proceed to NOFA.

This is a limited SI, which only uses a very small number of samples. Such samples are taken from the most likely locations of contamination. Therefore, each sample result will be compared to screening criteria.

3.6 Step 6: Specify Tolerable Limits on Decision Error – Define the decision maker's tolerable decision error rates based on a consideration of the consequence of making an incorrect decision.

3.6.1 Determine the Possible Range of the Parameter of Interest – Previous samples have not been taken at the former PAWL site. Therefore, the minimum value used for the parameter of interest is the concentration at the detection limit for each COC. The maximum values are those that exceed the appropriate screening criteria.

3.6.2 Identify the Decision Errors and Choose the Null Hypothesis – A non-statistical sampling plan will be used to position the location of each sample along with the number of samples. This process is being implemented because this is a limited SI. The sampling will be both purposeful and biased to locations that are most likely contaminated. Four steps are used to define where each decision error occurs relative to an action level and establish the decision errors associated with the sampling design.

Definition of Decision Error – As in any statistical test, there are two kinds of error that can occur in implementing the decision rule: the null

hypothesis may be rejected when true (Type I error, with probability α), or the fail to reject decision may be made when false (Type II error, with probability β). The number of samples needed to make the decision is driven by the error rate that can be tolerated, as well as by other considerations such as spatial variability of COCs distributions. Because this is a limited SI, probability errors will not be considered herein.

The Null Hypothesis (baseline condition) and the Alternative Hypothesis—The baseline conditions or null hypothesis for the PAWL is “COCs detected in the soil or surface water are at concentrations that warrant additional investigation activities”. The alternate hypothesis is “COCs detected in the soil or groundwater are at concentrations that do not demand additional investigation activities”. In terms of this investigation, the default assumption (null hypothesis) is that the concentrations of COCs at the site are significant enough to require further investigation.

Potential Consequence of Each Decision Error – In the event that the test results do not exceed the criteria levels when actual site levels do exceed criteria levels (Type I error), then possible endangerment of human health and the environment could occur. If this takes place then the Army Corps of Engineers and its sister agencies responsible for protection of human health and the environment would not be meeting their stated mission. If the COCs are detected at a concentration level that exceeds the criteria levels when actual site conditions do not, then the Army will have unnecessary expenses associated with additional investigation activities.

Which Decision Error Has More Severe Consequences Near the Action Level – For the purpose of protecting human health and the environment, the Type I error has more severe consequences in terms of the stated null hypothesis. The Type I decision error has a more severe consequence near the action level since the risk of jeopardizing human health is likely to outweigh the consequences associated with additional investigation expenditures and schedule delays. In using the data collected, careful review will be made to insure that the Type I error is not likely to occur. If the review warrants, additional investigation will be recommended.

3.7 Step 7: Optimization of the Sampling Design – The purpose of this step is to identify a resource-effective data collection design for generating data that are expected to satisfy DQOs. This SI will be based on surface and subsurface soil samples collected within and below the potential source area (that is, the former lagoons, themselves). As

more information is obtained, the sampling design may be optimized to accomplish the goals of this investigation.

No existing chemical data is available for the PAWL. Therefore, the sampling design is based on hypothetical expectations of what may be detected at the site. Alternative sampling plans could require additional samples and funds; however, the scope of this SI is limited in nature.

A review of historical information and reports indicated that a layer of 3 to 10 feet of fill material may exist over the former lagoons. Therefore, mechanical drilling equipment will be utilized to obtain subsurface soil samples.

The soils will be investigated by advancing 5 to 7 borings within the former waste lagoons. The borings will be advanced using a direct-push hydraulic sampler. Continuous soil samples will be obtained in 2 to 4-foot intervals until the boring termination depth is achieved or refusal is encountered. Boring termination depths will vary from 5 to 25 feet depending on observations at the time of the borings. We do not know if the lagoon's walls and bottoms were removed during decommissioning activities; therefore, it is possible they are still in place and may be impenetrable to our drilling equipment. Based on previous borings in the vicinity, bedrock refusal will be encountered at about 25 feet bgs. Proposed boring locations are given on the Boring Location Plan (see Field Sampling Plan (FSP)).

Field headspace screening will be performed on a portion of each sample interval based on visual indications. Headspace screening will be performed using a Photoionization Detector (PID). Field TNT screening will be performed on a representative portion of each sample interval. Screening for TNT will be performed using an EnSys Test Kit in accordance with the procedures outlined in the FSP. Each sample collected will be classified in accordance with the Unified Soil Classification System (USCS) using field index tests.

Selected soil samples will be sent, under chain of custody protocol, to an off site laboratory for chemical analysis. Chemical analysis of each laboratory sample will include VOCs, TAL metals, and Explosives. Testing for pH, TOC and Sieve/plasticity analysis will be performed for every change of soil strata. Samples selected for chemical analysis will be based on the interval most likely to be contaminated from the organoleptic indications and/or field screening results. Selection of samples for sieve/plasticity testing will be determined in the field in order to validate field USCS classifications.