U.S. ARMY CORPS
OF ENGINEERS
LOUISVILLE DISTRICT

Quality Assurance Project Plan

Limited Site Investigation of the former

Plum Brook Ordnance Works

Ash Pits Numbers 1 and 3

Sandusky, Ohio

DERP-FUDS HTRW Project No. G05OH001821

July 2000

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ATTACHED DOCUMENTS

I. Field Sampling Plan (FSP) dated May 1999
II. Data Quality Objectives (DQOs) dated May 1999

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, Liability Act of 1980
CERCLIS	Comprehensive Environmental Response, Compensation, and Liability Information System
CLP	Contract Laboratory Program
COC	Chain of Custody
D&D	Decontamination & Decommissioning
DERP	Defense Environmental Restoration Program
DNT	dinitrotoluene
DOD	U.S. Department of Defense
DQO	Data Quality Objective
EPA	U.S. Environmental Protection Agency
ERIIS	Environmental Risk Information & Imaging Services
FSP	Field Sampling Plan
FUDS	Formerly Used Defense Sites
GSA	General Services Administration
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 Lists
OEPA	Ohio Environmental Protection Agency
PA	Preliminary Assessment
PBOW	Plum Brook Ordnance Works
PBRF	Plum Brook Reactor Facility
PBS	Plum Brook Station
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
SI	Sight 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	U.S. Army Corp of Engineers
USCS	Unified Soil Classification System
USEPA	U.S. Environmental Protection Agency
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

SECTION 1

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 ash pits 1 and 3 located at the former Plum Brook Ordnance Works (PBOW). Located near Sandusky Ohio, the former PBOW was operated from 1941 to 1945 by the Department of Defense (DOD) as a manufacturing plant for trinitrotoluene (TNT), dinitrotoluene (DNT), and pentolite. Contamination detected at the site by several studies has been related to past activities and are being addressed under the Defense Environmental Restoration Program (DERP), Formerly Used Defense Sites (FUDS). The potential for contamination at the former ash pits 1 and 3 was identified in an Inventory Project Request (INPR) (Huntington District U.S. Army Corps of Engineers (USACE), 1998). A limited Site Investigation (SI) of these two ash pits will be conducted to evaluate the potential for contamination that may have resulted from past DOD activities.

1.2 Site Description

According to the Archive Search Report (USACE, 1993) land for the original site was acquired by Department of War in 1938 and consisted of approximately 9,010 acres of land. In the early 1940s the U.S. Army contracted with Trojan Powder Company to manufacture 2,4,6-TNT, dinitrotoluene and pentolite at PBOW. Production began on 16 December 1941 and continued through late 1945, ceasing two weeks after V-J Day (2 September 1945). After operations ceased, the area was turned over to Army Ordnance Department and renamed Plum Brook Depot and used for ammunition storage.

PBOW was placed in standby status from 1945 to 1946. During this time, decontamination and decommissioning (D&D) procedures were performed on many structures associated with the manufacturing process. D&D included the removal and relocation of all explosives to burn grounds for destruction by open burning. Where possible, remaining structures and buildings were burned in place. Drain lines and steam lines were flushed and dismantled. In December 1945, custody of PBOW was transferred from Trojan Powder Company to U.S. Army Ordnance Department, with the U.S. Army Corps of Engineers assuming custodial responsibilities from 1 January through 30 June 1946. In August 1946, PBOW was transferred to the War Assets Administration. In 1956 the National Advisory Committee for Aeronautics (NACA) began leasing sections of PBOW from the War Assets Administration. An agreement was made in 1956 to lease 500 acres of in the northern portion of PBOW to construct and operate the Plum Brook Reactor Facility (PBRF). In 1958 NACA changed its name to National Aeronautics and Space Administration (NASA). By 1963, approximately 6,400 acres of PBOW had been acquired by NASA for various aerospace research activities. An additional 2,000 acres were acquired to serve as a buffer zone between the facility and the adjacent community. Research and test activities were conducted by NASA at PBS throughout the 1960s and early 1970s. NASA declared approximately 2,150 acres as excess in 1978. Forty-six acres of the excessed property is used by the Perkins Township Board of Education as a bus

transportation center. Much of the remaining excess property was reclaimed as farmland. NASA also excessed Parcel #59 to the General Services Administration (GSA) for subsequent disposal.

1.2.1 Installation/Site Size and Borders

The PBS is a 6,453.5-acre research facility located in Erie County, Ohio 4.7 miles south of Sandusky and 59 miles west of Cleveland. Although primarily in Perkins and Oxford Townships, the eastern edge of PBS extends into Huron and Milan Townships. PBS is bounded on the north by Bogart Road, on the east by U.S. Highway 250, and on the west by County Road 43. State Route 4 is also located to the west.

The geographic coordinates for the extreme boundaries of PBS are:

Northern Boundary Latitude: 41° 23' 39"
Southern Boundary Latitude: 41° 20' 04"
Eastern Boundary Longitude: 82° 38' 39"
Western Boundary Longitude: 82°43'12"

Both Ash Pits are approximately 1 acre in size. Ash pit 1 is located south of Maintenance Road and north of Powerhouse 1. The northern and southern boundaries of ash pit 1 are still somewhat defined by berms or rises in elevation. The northern and southern berms transverse in an east/west direction along Maintenance Road and Powerhouse 1 respectively. The eastern and western boundaries of the pit are not clearly defined by a berm or rise in elevation, but have been identified from historical aerial photographs and drawings. Ash pit 3 is located west of Ransom Road and south of Maintenance Road within the boundaries of the NASA K-site research facility (formerly Powerhouse 3). The eastern border of Ash Pit 3 is the K-site research facility building. The remaining borders to the north, south, and west are thick vegetation.

1.2.2 Topography

According to the United States Geological Survey (USGS) quadrangle 7.5, ash pit 1 is located primarily on a flat grassland area at an elevation of approximately 635 feet mean sea level (msl). The ash pit is located south of Maintenance Road and north of Powerhouse 1 (figure 2). Powerhouse 1 and associated railroad tracks are still present at the site. The former ash pit is now covered with a thicket of grass, weeds, and small trees. The northern and southern boundaries of the ash pit are still somewhat defined by berms or rises in elevation approximately 3 to 5 feet. The northern and southern berms transverse in an east/west direction along Maintenance Road and Powerhouse 1 respectively. Both of these berms are approximately 640 feet msl. The eastern and western boundaries of the pit are not clearly defined by a berm or rise in elevation, but have been identified from historical aerial photographs and drawings. A drainage ditch is located on the northern boundary of the ash pit and runs in a northerly direction and under Maintenance Road by means of a concrete culvert.

Ash pit 3 is primarily located on a flat grassland area at an elevation of approximately 635 feet msl. The ash pit is located west of Ransom Road and south of Maintenance Road (figure 3). The Powerhouse 3 area has been reconfigured by NASA and is now a research test facility (K-site). The surrounding area around the ash pit is at an elevation of 640 feet msl. The K-site test facility is

currently supplying non-contact cooling water to the former ash pit, which is now holding water. The depth of the water in the pit has not been ascertained. A pipe at the northeastern corner of the pit is supplying the cooling water. It is assumed that this pipe was the original pipe that once supplied the ash to the pit. The boundaries of the pit appear to be the same as the original boundaries, with the exception of an indention to the west on the eastern boundary. Railroad tracks running in a north-south direction are still present at the site. The pit is partially surrounded by mature and smaller trees. The water is discharged from the former pit by means of an east-west trending surface water ditch that eventually discharges into Pipe Creek.

1.2.3 Geography & Hydrogeology

Two formations of the Devonian Age underlay both ash pits 1 and 3. The oldest formation is the Delaware Limestone, which is characterized as a hard, dense, finely crystalline limestone and dolomite. Dissolution of this unit has produced solution channels along bedding planes and joints, and even producing caverns in some areas. The unit is typically buff colored and is fossiliferous. Overlaying the Delaware Limestone is the Olentangy Formation. This formation is made up of two members, the Plum Brook Shale and the overlying Prout Limestone. Only the Plum Brook Shale member should be encountered at the ash pits. 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 Olentangy formation is the first natural boundary beneath the ash pits at an approximate depth of 25 feet below ground surface (IT, 1999).

The overburden soils at both ash pits is predominately composed of glacial till, outwash, or lacustrine (lake) deposits. It is assumed that the overburden at both pits is approximately 25 feet thick (IT, 1999). The surface soil at both sites is of the Kibbie-Elnora-Tuscola-Colwood Association (ODNR, 1994). This association is described as nearly level to gently sloping. The soil is described as somewhat poorly drained, moderately well drained, and very poorly drained formed in outwash, lucrastine, and deltaic sediments. Based on review of boring logs advanced in the vicinity of the former ash pits, overburden soils generally consist of an upper layer of silty fine sand and silt of low plasticity. These materials typically are classified as SM and ML, respectively, in accordance with the Unified Soil Classification System (USCS). This upper layer is generally underlain by silty clay of low to high plasticity extending to bedrock. This material was typically classified as CL or CH in accordance with the USCS (IT, 1999).

Based on a review of the site wide groundwater study (IT, 1999), groundwater is encountered in the bedrock units underlying the ash pit sites. Generally the groundwater flows northward toward Lake Erie. Groundwater in the overburden soils is generally expected to be purge or trapped water. Purge water occurs in irregular, discontinuous granular zones within the soil overburden. Purge water sources contain widely varying quantities of water depending on recent precipitation. Based on expected soil types, overburden groundwater quantities if encountered, should be minimal.

1.3 Past Data Collection Activities

In March 1995, Dames & Moore conducted a field reconnaissance of the area north of Power Station 1. A depression was observed between the railroad tracks and Maintenance Road. The depression contained small trees, grasses, and some bare areas having black gritty soil. The location of this

depression corresponds to the location of ash pit 1 in historical drawings and aerial photographs. No surface water, sediment, surface soil, subsurface soil, or ground water samples have been collected at ash pit 1.

Ash pit 3 is located at what is now NASA's K-site test facility, west of Ransom Road and south of Maintenance Road. During a field reconnaissance of this area in the fall of 1994, representatives of Dames & Moore and CELRN observed a surface water impoundment west of the NASA K site building formerly powerhouse 3. Water flowed into the impoundment through a steel pipe at its northeast corner, and water discharged from impoundment into an east-west trending surface water ditch, which discharges into Pipe Creek. The impoundment corresponds to the location of ash pit 3 that appears in historical drawings and aerial photographs. No surface water, sediment, surface soil, subsurface soil, or ground water samples have been collected at ash pit 3.

1.4 Current Status

The locations of the subject ash pits are represented in figures 2 and 3. During the period when PBOW was operational, DoD constructed and utilized 3 power stations corresponding to the power and maintenance areas 1, 2, and 3. Each power station consisted of a main powerhouse, a coal storage area, and a diked area containing fuel oil storage tanks (ASTs). Each power house building consisted of a boiler house, compressor room, electrical room, filter room, and locker room. The buildings also contained two to four large coal-burning boilers, a turboelectric generator, a feed water treatment system, and several steam driven or electric air compressors. The generated steam was used for space heating, driving compressors, and generating electrical power. Fly ash generated from each of the boilers in the powerhouse collected in pits. Water was added to the ash and the water ash slurry flowed through a sluice trench to the ash sump located at the end of the building. From the ash sump, the ash traveled through a pipeline to a nearby surface water impoundment (ash pit) (D&M, 1995). Information concerning the status or use of the ash pits following the war is limited. During the 1960's, the power plant facility at power maintenance Area 3 was remodeled to become the K-Site Test Facility (NASA-PBS) (MK 1994a). NASA is currently using K-Site to perform research.

1.5 Project Objectives

The purpose of this limited Site Investigation (SI) is to evaluate the potential for contamination of the site due to past DoD 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 analyses 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, if warranted by analytical results, is beyond the scope of this project and will be performed in subsequent phases.

The field investigation for this limited SI will include:

- A visual survey to assess and document current site conditions.
- Surface and subsurface soil sampling.

- Sediment and surface water sampling.

1.5.1 Project Target Parameters and Intended Data Usages

Target parameters include Semi-volatile organic compounds (SVOCs) and Target Analyte List (TAL) metals. These parameters will be identified based on analytical results using 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 RPGs).

Geotechnical and additional environmental data will be gathered to help assess the physical and potential migration characteristics of the subject sites. This includes pH, total organic carbon (TOC), soil grain size distribution, in accordance with ASTM D422, and soil plasticity (Atteberg Limits) in accordance with ASTM D4318.

1.5.2 Quality Objectives and Criteria for Measurement Data

Quality objectives and criteria for this limited SI were evaluated through the Data Quality Objective (DQO) Process described in the United States Environmental Protection Agency's (USEPA) QA/G4 (September 1994) guidance document. The DQO process is a series of planning steps based on the scientific method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are logical and appropriate for the intended application. The DQO process for this site investigation is formally documented and attached to this QAPP.

Sample Network Design and Rationale

The attached FSP and DQOs describe the rationale used to select sampling locations and depths.

1.6.2 Site Maps of Sampling Locations

Maps showing intended soil, sediment and surface water sampling locations are included as figures 2 and 3.

1.7 Project Schedule

The earliest date for which field activities will begin is June 1999.

SECTION 2

PROJECT ORGANIZATION AND RESPONSIBILITY

This work is being performed 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 Scientist:	Darrell Davis, CELRL-ED-EB
Safety QC:	Shirley Dunn, CELRL-SO
Risk Assessor:	David Brancato, CELRL-ED-EE
Hydrogeologist:	Martin Wahking, CELRL-ED-EB
QA Manager/Chemist:	Samir Mansy, CELRL-ED-EB
Technical Manager:	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 has the additional training requirements specified by OSHA in 29CFR1910.120. She will serve as the QC reviewer and alternate 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

Project Manager

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

Technical Manager

Doug Meadors serves as the Technical Manager (TM) has overall responsibility for ensuring that the projects 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.

Project Scientist

Darrell Davis serves as the Project Scientist (PS) and is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The PS primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The PS 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 USACE resources as needed to ensure performance within budget and schedule constraints.
- Orient all field teams and support staff concerning the project's special considerations.
- Monitor and direct the field teams
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task project.
- Review the work performed on each task to ensure its quality, responsiveness and timeliness.
- Review and analyze overall task performance with respect to planned requirements and authorizations.

- Prepare all reports (deliverables) for submission to Ohio EPA.

- Ultimately be responsible for the preparation and quality of interim and final reports.

2.3 Quality Assurance (QA) responsibilities

QA Manager

The 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, USA Corps of Engineers, and Ohio EPA requirements. Specific functions and duties include:

- Providing QA audit on various phases of the field operations;

- Reviewing and approving of QA plans and procedures;

- Providing QA technical assistance to project staff;

- Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the program manager and to the technical manager, Doug Meadors.

- Data validation including tentatively identified compounds;

- Review and approval of field, and laboratory procedure.

- Performance and system Audits of the Laboratory.

2.4 Field Responsibilities

Field Team Leader

He/she is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his/her supervision. The field team leader is an environmental professional and will report directly to TM. For this investigation the project scientist will serve as the field team leader. 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.
- Conducting daily safety meetings

Field Technical Staff

The field technical staff (team members) for this project will be drawn from the Louisville District, Environmental Engineering Branch. The technical field team staff will be utilized to gather information, collect samples, and to prepare various task reports and support materials. All of the designated technical team members are experienced environmental professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required field activities.

2.5 Laboratory Responsibilities

Quanterra Laboratory Project Manager

The Quanaterra project manager, Debora Hula, will report directly to the USACOE 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;

Over viewing 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 Project 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 that they 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 located in appendix B.

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 obtained from all the measurements taken in the project. The equation for completeness is presented in section 12 of the QAPP. Field completeness for this project will be greater than 90 percent.

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 field sampling plan (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 Field Sampling Plan (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 are 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.

The QC level of effort for the field measurement of pH consists of pre-measurement calibration and post-measurement calibration and post-measurement verification using two standard reference solutions each time as appropriate to the sample tested. The QC effort for field conductivity measurements will include daily calibration of the instrument using standard solutions of known conductivity.

SECTION 4

SAMPLING PROCEDURES

A detailed Field Sampling Plan was developed and used for this site investigation and is consistent for the purpose of this project. The field sampling plan outlines all the sampling procedure information and is attached to this document.

SECTION 5

CUSTODY PROCEDURES

Custody is one of several factors which is 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 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

Detailed information on field custody procedures can be found in attachment I containing the Field Sampling Plan.

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 laboratory 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 which constitute evidence relevant to sampling and analysis activities as described in this QAPP. The PS 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 PS.

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

- progress reports, QA reports, interim project reports, etc.

- 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

Field screening and associated field instruments were not required for this site investigation.

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.

After the tuning criteria are met, the instrument is initially calibrated using a five point calibration curve. 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

Field screening and associated field instruments were not required for this site investigation.

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 (VOA) shall be screened in the laboratory, as described in the VOA 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 site soil sample extracts requiring pesticide/PCB and/or semivolatile organic compounds analysis (acid/base/neutral analysis or ABNs) shall be subject to gel permeation chromatography cleanup and/or other column chromatography cleanup as necessary.

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.

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 figure () 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

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 applicable procedures and at the frequency indicated can be located in the Field Sampling Plan (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
- Endrin/DDT degradation checks for GC/EC analysis
- Second dissimilar column confirmation for GC/EC 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 these procedures which are 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. Only direct reading instrumentation will be employed in the field. The use of pH meters, thermometers, an OVA, and a probe to measure specific conductance will generate some measurements directly read from the meters following calibration per manufacturer's recommendations as outlined in Section 6 of this QAPP. 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. Later, when the results forms required for this study are being filled out, the field manager, identified in Section 2 of this QAPP, will proof the forms to determine whether any transcription errors have been made by the field crew.

Because the use of field instrumentation such as a mobile gas chromatograph will not be used until a later phase of the study has been reached, there will be no further need for assuring that field data has been reduced properly through the use of formulas or interpretation of raw data printouts.

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 format. 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 quanitation, tentatively identified compounds, 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. Ten percent (10%) of the data shall be 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: 1) concentration below required detection limit, 2) estimated concentration due to poor spike recovery, and 3) concentration of chemical also found in laboratory blank. The Data Reviewer comments will indicate that the data are: 1) useable as a quantitative concentration, 2) 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 activities.

9.3.2 Laboratory Data Reporting

The task of reporting laboratory data the U. S. A . Corps of Engineers begins 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 analysis 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.A. Corps of Engineers. The U.S.A. 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.A. 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 Frequency

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.A. 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 Preventative Maintenance

No field instruments will be used for this site investigation.

11.2 Laboratory Instrument Preventative Maintenance

As part of their QA/QC Program, a routine preventative maintenance program is conducted by a service contractor on a limited basis. Quanterra Laboratories 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:

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

Control Charts for recoveries (%), and RPDs will be submitted with the data packages to the USA 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 encounter 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 Scientist. If immediate corrective action is required, approvals secured by telephone from the Project Scientist 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 project manager, who in turn will notify the USACE 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 USA 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 or the Project Scientist.

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 field team leader or designee. This manager 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 warrant's a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the manager.

The manager 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 field team leader 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 operations manager and field

team. Corrective action will be documented in quality assurance reports to the entire project management.

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 USACE 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 are 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 holding time criteria 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, PM 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 refereed 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 USA 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 USA Corps of Engineers data assessor identifies a corrective action situation, it is the project manager who 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 deliverables associated with the tasks identified in the Work plan and monthly progress reports will contain separate QA sections in which data quality information collected during the task is summarized. Those reports will be the responsibility of the project manager and will include the Quality Assurance Officer report 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

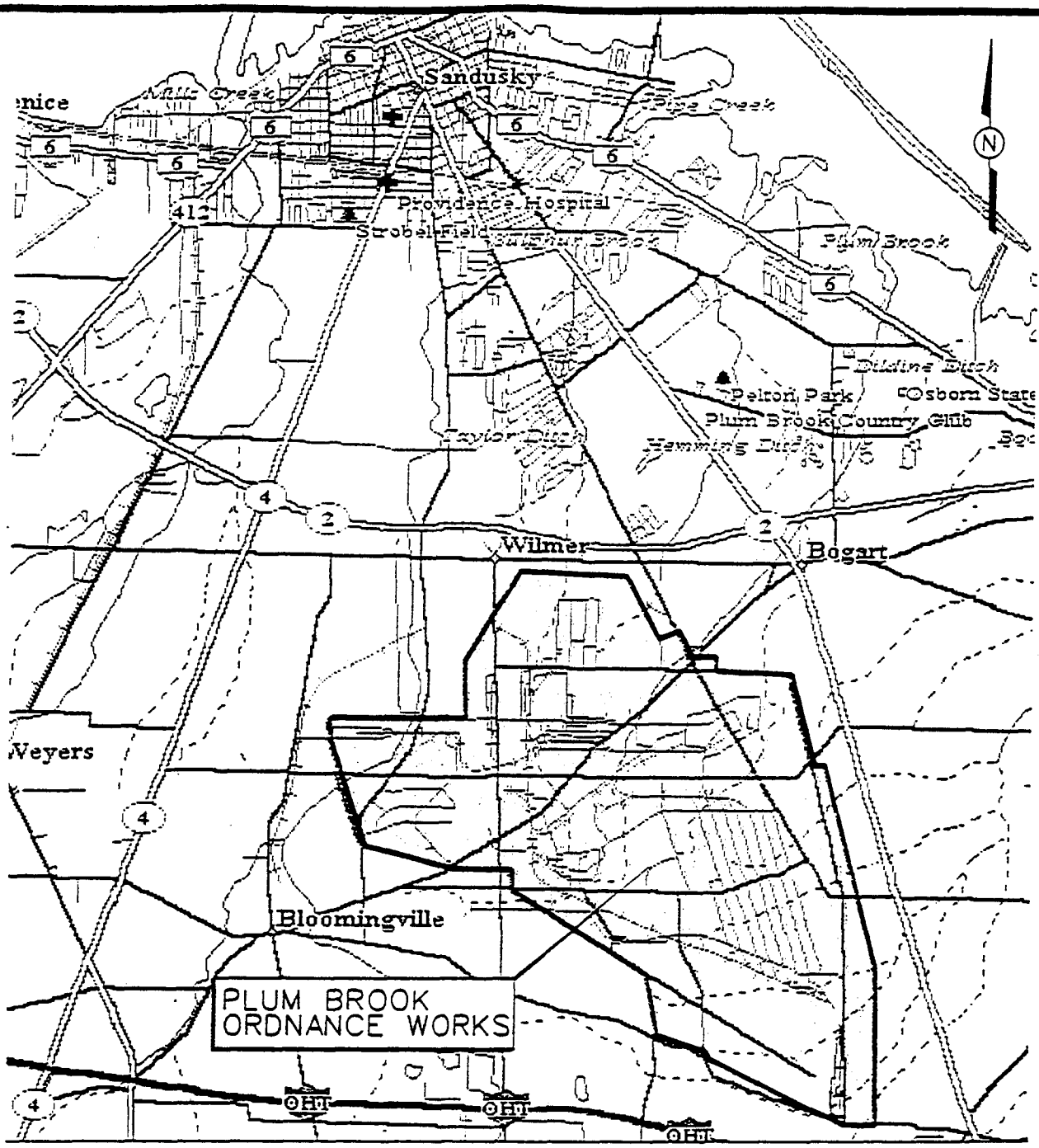
The QA reports will contain on a routine basis all results of field and laboratory audits, all information generated during the past month reflecting on the achievement of specific data quality objectives, and a summary of corrective action that was implemented, and its immediate results on the project. 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 reports will be prepared in written, final format by the 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 next issue of the monthly QA report.

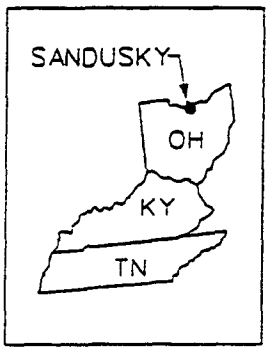
14.2 Frequency of QA Reports

The QA Reports will be prepared on a monthly basis, and will be delivered to all recipients by the end of the first full week of the month. The reports 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

All individuals identified in the Project Organization chart will receive copies of the monthly QA report.

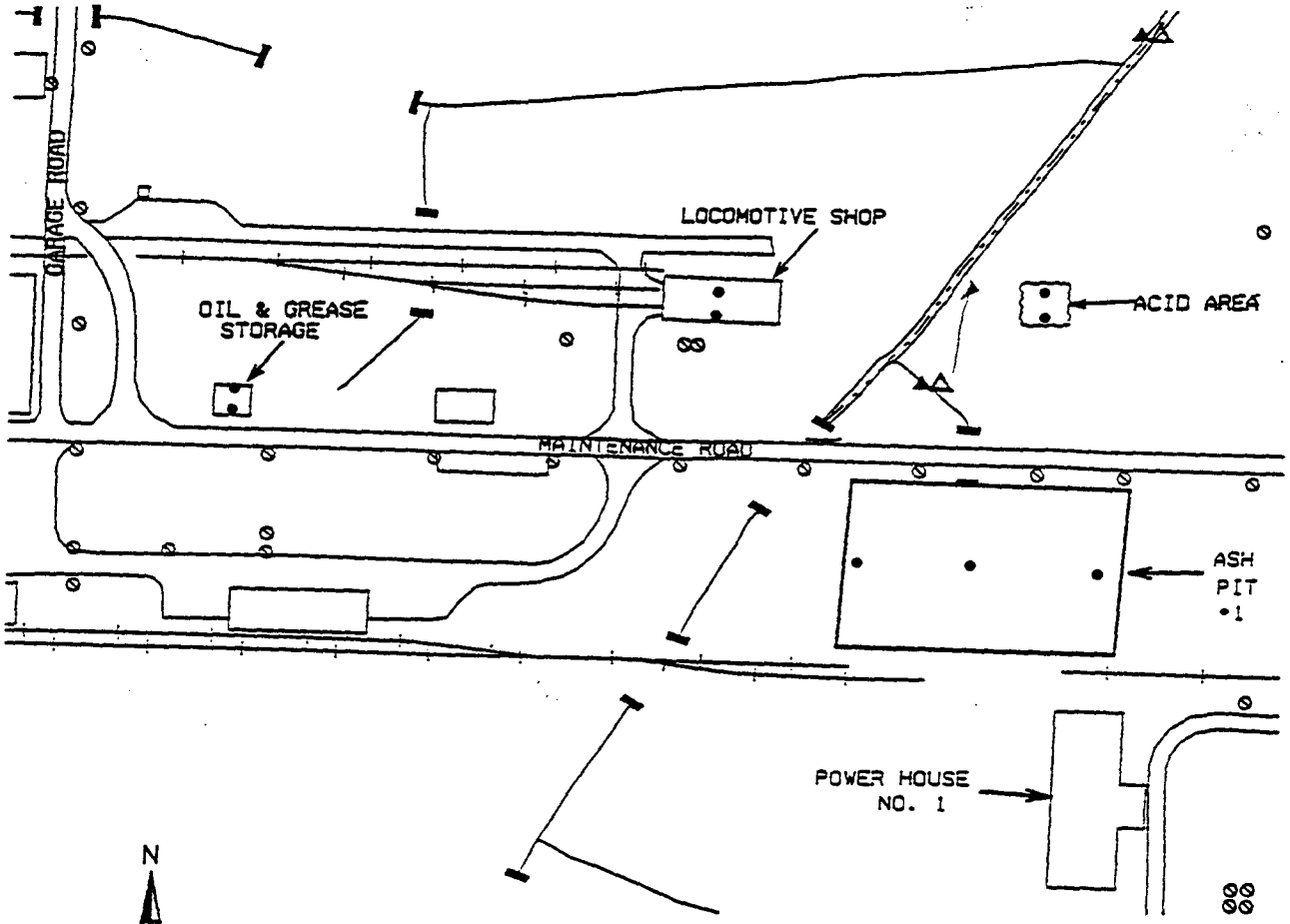




NOT TO SCALE

PLUM BROOK ORDNANCE WORKS
SANDUSKY, OHIO

FIGURE 1



N

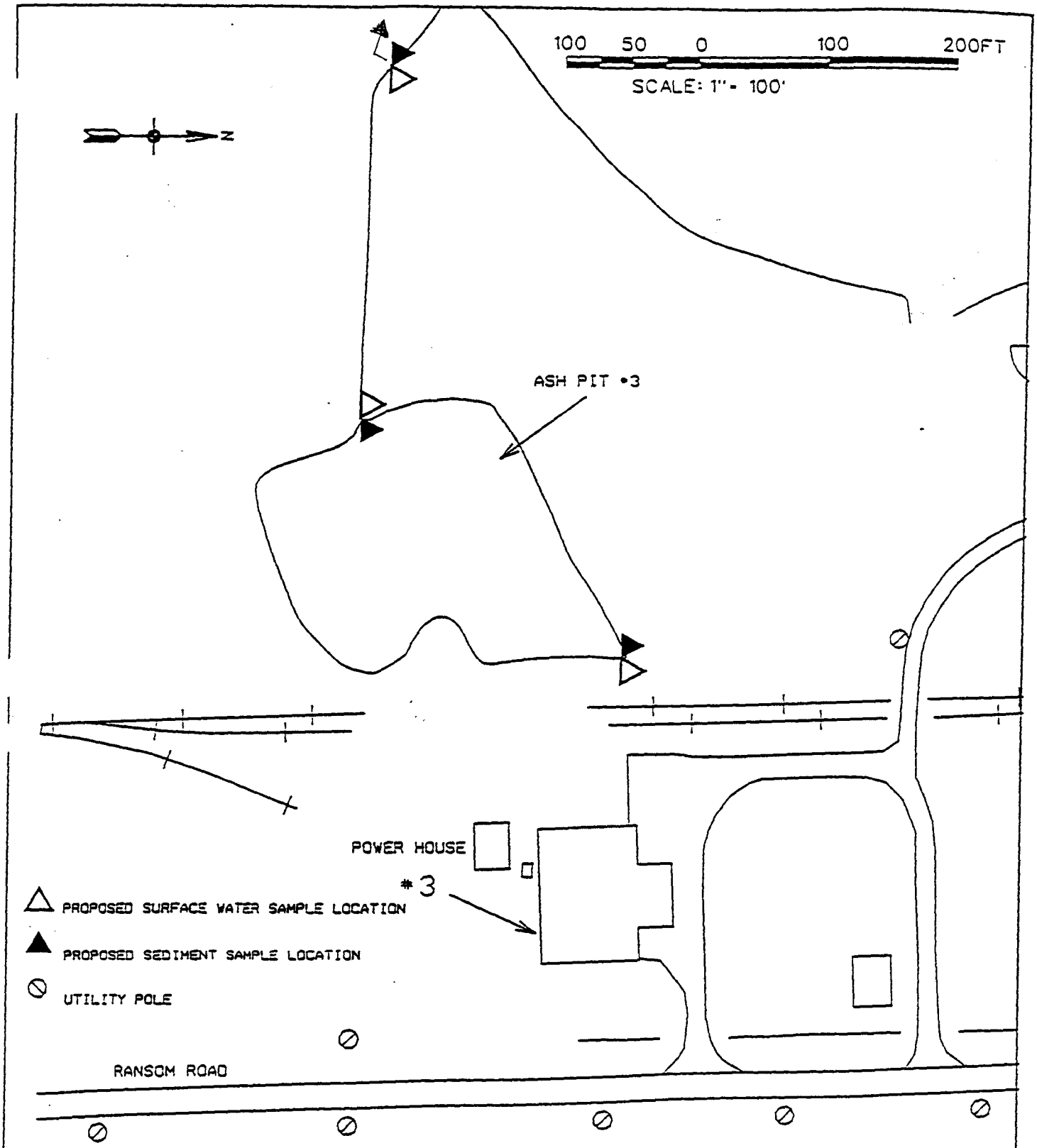
200 100 0 200 400FT

SCALE: 1" = 200'

- ● PROPOSED BORING LOCATION
- △ PROPOSED SURFACE WATER SAMPLE LOCATION
- ▲ PROPOSED SEDIMENT SAMPLE LOCATION
- ⊘ UTILITY POLE
- ▬ CULVERT (SYMBOL)

Design By:	Drawn By:	Checked By:	US Army Corps of Engineers	PLUM BROOK STATION SANDUSKY, OHIO LIMITED SITE INVESTIGATION FY 99	PROPOSED SAMPLE LOCATIONS ASH PIT #1 GARAGE MAINTENANCE AREA
Reviewed By:	Approved By:				
Date:	Scale: 1" = 200'	Drawing Code:	Sheet Ref. No.	Sht 1 of 7	

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 ASH PIT #3
Reviewed By:	Approved By:				
Date:	Scale: 1" = 100'		Drawing Code:	Sheet Ref. No.	Sht 2 of 7

FIGURE 3

APPENDIX A

ETHICS AGREEMENTS

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Debora 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, Stacey 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.

Stacey 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.

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.

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.

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.

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. 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. I, Patty 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.

Patty McClain

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. 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)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. I, Michael Stamper *(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 Stamper

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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)*:

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, 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.

Dorothy J. Leeson

(Signature)

April 20, 1999

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, [signature] *(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]

(Signature)

4/20/95

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. I, Melissa A. Fuller-Gustavel *(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-Gustavel

(Signature)

20 April 1999

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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)*:

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 A Macenczak

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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 Quanterra *(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.

Tanesha A. Roberts

(Signature)

4-20-99

(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. 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)*:

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.

Heather M Bosworth

(Signature)

4.20.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Scott Zomer *(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, 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. 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. 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. I, Brian 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.

Brian M. Greenwell

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Kim D. 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 D. Davis

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Carolyne 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.

Carolyne 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. 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 J Dempster

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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.

Rhonda Kuster

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Bradley Belding *(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.

Bradley E. Belding

(Signature)

4/21/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. I, Jeffrey 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.

(Signature)

April 20, 1999

(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)

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.

(Signature)

4/26/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. 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. 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;

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)

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. 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;

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.

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 Ulman

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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;

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, 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.

Tammy Tokos

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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.

(Signature)

4/21/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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, Donny 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.

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, LEE 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 Mancine *(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.

Louis Mancine

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Sharon Gei *(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 Gei

(Signature)

4-21-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. 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.

(Signature)

4-21-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, JON W. RITTENOUR *(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. I, Chris Grandi *(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 Grandi

(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 Kardohely *(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.

Sue Kardohely

(Signature)

4.20.99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. I, Kevin McDaniel *(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 J McDaniel

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. I, LESLIE M. VANKUREN *(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. 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;

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.

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. McCrory *(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. McCrory

(Signature)

4-20-99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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;

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 E. Stiller

(Signature)

4/20/99

(Date)

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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;

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, 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. 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. I, Tami 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.

Tami Stephens

(Signature)

4-20-99

(Date)

K.M. HUTRAS

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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)

R. Ly

Appendix E

Example Ethics Agreement

Quanterra, Inc.

(Laboratory Name)

ETHICS AND DATA INTEGRITY AGREEMENT

I. 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. 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. 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)

APPENDIX B

QUANTERRA STANDARD OPERATING PROCEDURES (SOPs)

QUANTERR. NORTH CANTON

Laboratory Director
Chris Oprandi

Customer Service & Sales
Robert McGregor

- Project Management
  - Alesia Danford, PM
  - Dave Heakin, PM
  - Roger Toth, PM
  - Ken Kuzior, PM
  - Denise Pohl, PM
  - Jeff Smith, PM
  - Becki Strait, PM
  - Amy McCormick, PM
  - Gary Wood, PM
- Project Administrators
  - Kris Buzash, PA
  - Michele Daley, PA
  - Doris Herren, PA
  - Barb Girard, PA
  - Pat Blimm, Recept (P)
  - Ari Blakeman, Recept (P)
- Customer Service DOD - Deb Hula, CSM/Sr PM
- FAS, Will Cordell, Supervisor
  - Joe Grant, Technician
- Sales
  - Jay Bernarding
  - Pat Mc Issac
  - Barb Hall
  - Rick Carr

Operations Support

- Mark Bruce, Technical Advisor
- Steve Jackson, Env. Health & Safety
- Lisa Haueter, Administrative Asst (P)
- Jim Horton, LAN Analyst
  - Susan Palmer, Lims Administrator

Lab Supervisor
Stacey Campbell

- MS Semivolatiles, Tom Hula, GL
  - John Gruber, Analyst
  - Tammy Tokos, Pkg Gen
  - Mark Ulman, Analyst
- GC/Organic Prep, Ray Risden, GL
  - GC Semivolatiles
    - Kim Davis, Data Entry
    - Sue Dempster, Analyst (P)
    - Rhonda Kuster, Analyst
    - Carolynne Roach, Analyst
    - Paul Sharkey, Analyst
    - Riley Salomns, Analyst
    - Kim Hutras, Pkg. Gen.
    - Brian Greenwell, Analyst
  - Extractions
    - Lou Mancine, Analyst
    - Tom Fausnight, Analyst
    - Lee Nelmes, Analyst
    - Donnie Williamson, Analyst
    - Eric Miller, Analyst
    - Cooper Walsh, Analyst (P)
    - Jeff Austin, Analyst (P)
    - Sharon Gels, Analyst (P)
- Volatiles, Tom Stiller, GL
  - MS Volatiles
    - Sherry Arman, Pkg Gen
    - Steven Macenczak, Analyst
    - Laura Evans, Analyst
    - Sue Kardohely, Analyst
    - Jennifer McCrory, Analyst
    - Kevin McDaniels, Analyst
    - Rick Qualye, Analyst
  - GC Volatiles
    - Leslie VanKuren, Analyst
    - Angel Siegfried, Analyst
    - Mike Horvath, Analyst
- Shipping, Al Haidet, Supervisor
  - Greg Newman, Tech
- Sample Mgmt, Lois Ezzo, GL
  - Terry Burns, Tech
  - Tami Stephens, Tech
  - Jennifer Girard, Tech
- Maintenance, Brian Haueter, Supervisor
  - Jason Umbaugh, Tech
- Metals, Patrick O'Meara, GL
  - Melissa Cordell, Analyst
  - Karen Counts, Analyst
  - Al DiPofi, Analyst (P)
  - Heather Boswell, Analyst
  - Lisa McGall, Data Entry
  - Melissa Recznik, Analyst (P)
  - Larry Williams, Analyst
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March 31, 1999

QUANTERRA® STANDARD OPERATING PROCEDURE

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

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

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1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of 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-µm 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 µL 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 µL, 20 µL of a 400 ug/mL internal standard solution would be added for a 1 µL injection.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 10 µg/mL or 50

µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 10 µg/mL or 50 µg/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 ≤ 6°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 ≤ 6°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 ± 2°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 +/- 3 standard deviations for surrogates, MS and LCSPrecision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations.

9.2.1 These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is 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 repreparation 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/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 $\geq$ 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/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 form 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 preceeding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 40 ng injected on column (for example, 8 µL or 50 µL internal standard solution in 0.5 mL of extract for a 1 µL 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 ± 2°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 preceeding 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 ±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 ±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

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

RF = Mean of RFs from intial caibration for a compound

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

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

RFi = 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 ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

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

12.5.3.2 Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C= Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000

µL. 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:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

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 (µg/L)

C_{is} =Concentration of the specific internal standard (µg/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 be 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 1

Quanterra Primary Standard[1] 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 Standard[1] 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 IX[1] 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
Phenazine	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1600
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Dialifate	2303-16-4	20	660
Dimethoate	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	298-04-4	50	1600
2-sec-butyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
*Methyl Parathion	298-00-0	50	1600

Table 2

Quanterra Appendix IX[1] 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
Diethylphthalate	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[4]	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
Butyl 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

[3] 2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[4] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. 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				
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	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp	3689-24-5				X
Phorate	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7				X
*Methyl parathion	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 µl
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)[1]	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/µL 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/µL if 2 µL injection is used

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL 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/µL if 2 µL 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 µL 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	150

[1] Included in standard mix, but not routinely evaluated for method 8270B

[2] Levels are 50 and 75 ng µL if 2 µL injection is used

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12[3]
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
Dimethylphthalate	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
Diethylphthalate	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 12[3]
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-diphenlyhydrazine. 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 13[1]

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 13[1]

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,

[2]Optional 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	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(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	60	31.5	41.5-130.6
d-BHC	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	64.9-114.4
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
4,4'-DDD	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	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	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 µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

[1]Since 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/µL 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/µL 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/µL if 2 µL 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))

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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 HNO_3, 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 achievably**, 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°C ± 4°C, or equivalent heating source

6.5. Calibrated Thermometer: 0°C - 200°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 (HNO_3), 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 HNO_3 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 HNO_3 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 HNO_3 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 regent 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 HNO_3 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

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

STANDARD DEFINITIONS

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		

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))

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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 HNO_3 and H_2O_2. For GFAA preparation, the digestate is refluxed with HNO_3. 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, HNO_3 Concentrated: Reagent grade

7.1.2. Hydrogen Peroxide, H_2O_2, 30%: Reagent grade

7.1.3. Hydrochloric Acid, HC1, 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 1.

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 HNO_3, cover with a watchglass, place the beaker on a hotplate at 91-99°C, and reflux 15 minutes **without boiling**. Add 5 mL concentrated HNO_3, replace watchglass and reflux for an additional 30 minutes. Add an additional 5 mL concentrated HNO_3 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% H_2O_2, **cover with watchglass,** and heat on hotplate. Once effervescing stops, add H_2O_2 in 1 mL increments to a total of 10 mL H_2O_2.

NOTE: Do Not add more than a total of 10 mL 30% H_2O_2

11.3.4. For FLAA and ICP preparation, add 5 mL concentrated HC1 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 HC1.

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 HC1 and 15% v/v HNO_3 (ICP/FLAA/SB). (GFAA 15% v/v HNO_3 - No HC1).

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					

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)

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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°C ± 2°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.

Client ID Sample Number	Assigned Laboratory Number
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 reprepped 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 ☐

13. Was a sufficient amount of sample sent in each bottle? Yes ☐ No ☐

Contacted PM _____ Date:_____ by:_____ via Voice Mail ☐ Verbal ☐ Other ☐

Concerning:_____

Check √	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		
√	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

Quanterra ® Cooler Receipt Form/Narrative

North Canton Facility

Client ID	pH	Date	Initials

Cooler	Temp	Method	Comments

Discrepancies Cont.

Macro Name:

Macro Name:

Macro Name:

Other Anomalies:

Preservative Preparation

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 $Zn(C_2H_2O_2) \bullet H_2O$ 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_2SO_4	250 mL
Chloride (Cl)	P	None	250 mL	Total Organic Halogens	G	H_2SO_4	250 mL
Chromium [illegible]	P	None	250 mL	COD	P	H_2SO_4	250 mL
R. Chlorine	P	None	100 mL	Ammonia Nitrogen (NH_3)	P	H_2SO_4	500 mL
Color	P	None	50 mL	TKN	P	H_2SO_4	1L
Conductivity	P	None	250 mL	Nitrate/Nitrite	P	H_2SO_4	250 mL
Corrosivity	P	None	250 mL	Oil & Grease	G	H_2SO_4	1 L
Dissolved Oxygen	G	None	300 mL	Phenols	G	H_2SO_4	1 L
Fecal Coliform	P	None	125 mL	Total Phosphorus	P	H_2SO_4	250 mL
Flashpoint		None	100 mL	TOX	P	H_2SO_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
PNA/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, HNO_3, and H_2SO_4 to pH < 2. NaOH to pH > 12

[2] Temperature = 4°C ± 2°C except for aqueous metals

[3] Samples to be anlayzed 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 Analysis Requisition

Example Subcontracted PO Logbook

APPENDIX C

LABORATORY ANALYSIS CRITERIA

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)	Instrument Evaluation: SPCCs: minimum RF values per method Requirements CCCs: verify RSD ≤ 30% Primary Evaluation (all target analytes) r ≥ 0.995, RSD ≤ 15%, r2 ≥ 0.990 Alternative Evaluation. Mean RSD for all target analytes ≤ 15%	No allowance Alternative Evaluation: Maximum allowable RSD for each target analyte ≤ 40%
ICV (9.3)	1. Recovery = 70 - 130% 2. **QC/MRL: D ≤ 20%**	Sporadic Marginal Failures': Recovery = 50 - 150%
CCV (9.5 / 9.5.2 9.5.2.4)	Instrument Evaluation: 1. SPCCs: minimum RF values per method requirements 2. CCCs: verify D ≤ 30% 3. Primary Evaluation (CCCs Drift ≤20%, D ≤ 20% 4. **QC/MRL: D ≤ 20%**	Primary Evaluation (remaining target analytes): Qualitative, see text
MB (10.2.1 / 11.4.1)	Target Analytes: Analytes < MDL Check Sample (-2X MDL)	Common Lab Contaminants: Analytes ≤ MDLs
LCS (10.2.2 / 11.4.2)	Water: Recovery = 60 - 120% (-15 analytes) = 45 - 135% (-30 analytes) = 20 - 150% (-45 analytes) Soil: Recovery = 60 - 120% (-20 analytes) = 45 - 135% (-25 analytes) = 30 - 150% (-45 analytes)	Sporadic Marginal Failures': Water: Recovery = 15 - 150% Soil: Recovery = 25 - 150%
MS (10.2.3 11.4.3 11.4.3.2)	Water: Recovery = 45 - 135% Soil: Recovery = 45% - 135%	Sporadic Marginal Failures': Water: Recovery = 15% - 150% Soil: Recovery = 20% - 150%
MSD/MD (10.2.4 / 11.4.4)	Water: RPD ≤ 50 Soil: RPD ≤ 60	Sporadic Marginal Failures'-: Water: RPD≤ 60 , Soil: RPD≤ 60
Surrogates (10.2.5 / 11.4.5)	Interference- Free Matrix: Water: Recovery = 60 - 120% B/N cmpds Recovery = 45 - 135% A cmpds Soil: Recovery = 60 - 120% B/N cmpds Recovery = 45 - 135% A cmpds Project Sample Matrix: Water: Recovery = 45 - 135% B/N cmpds Recovery = 35 - 140% A cmpds Soil: Recovery = 45 - 135% B/N cmpds Recovery = 35 - 140% A cmpds	Sporadic Marginal Failures': Water: Recovery = 15 - 150% Soil: Recovery = 20 - 150%

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 ≥ 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 ≥ 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 ≥ 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 stnd**	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 v 10%
Method of Standard Addition (MSA) (12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	r ≥ 0.995

APPENDIX D

DATA VALIDATION CHECKLIST

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 data7

[ ] 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/A

Note 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/I\~S 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 RR~ 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.

COMMENTS

The case description and exceptions, if any, are noted below, with reason(s) for rejection (R) or qualification as estimated (J). Any laboratory deficiencies are also noted in this section.

COMMENTS (Cont'd)

Reviewed By: _____

Name: _____

Concurred By: _____

Name: _____

APPENDIX E

INORGANIC DATA VALIDATION CHECKLIST

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%	[ ] Yes	[ ] No
Mercury	80 - 120%	[ ] Yes	[ ] No
Cyanide	85 - 115%	[ ] Yes	[ ] 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%, 1 16% - 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 e 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--wi1---~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?	[ ] Yes	[ ] No
Solid Samples?	[ ] Yes	[ ] 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.

COMMENTS

The case description and exceptions, if any, are noted below, with reason(s) for rejection (R) or qualification as estimated (J). Any laboratory deficiencies are also noted in this section.

COMMENTS (Cont'd)

Reviewed By: _____

Name: _____

Concurred By: _____

Name: _____

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				Check List 1660					Spike List 1661				
Compound	RL	Units	MDL	Units	Run Date	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				Check List 1660					Spike List 1661				
Compound	RL	Units	MDL	Units	Run Date	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: 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

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	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				Check List 904					Spike List 905				
Compound	RL	Units	MDL	Units	Run Date	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 Reference 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	mg/kg	70	130	20		0.1666	mg/kg	70	130	20	

Quanterra Reference 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: 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 (± 100%) associated with the results calculated at the MDL. The error associated with the MQL should be comparable to the CCV acceptance limits (± 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.

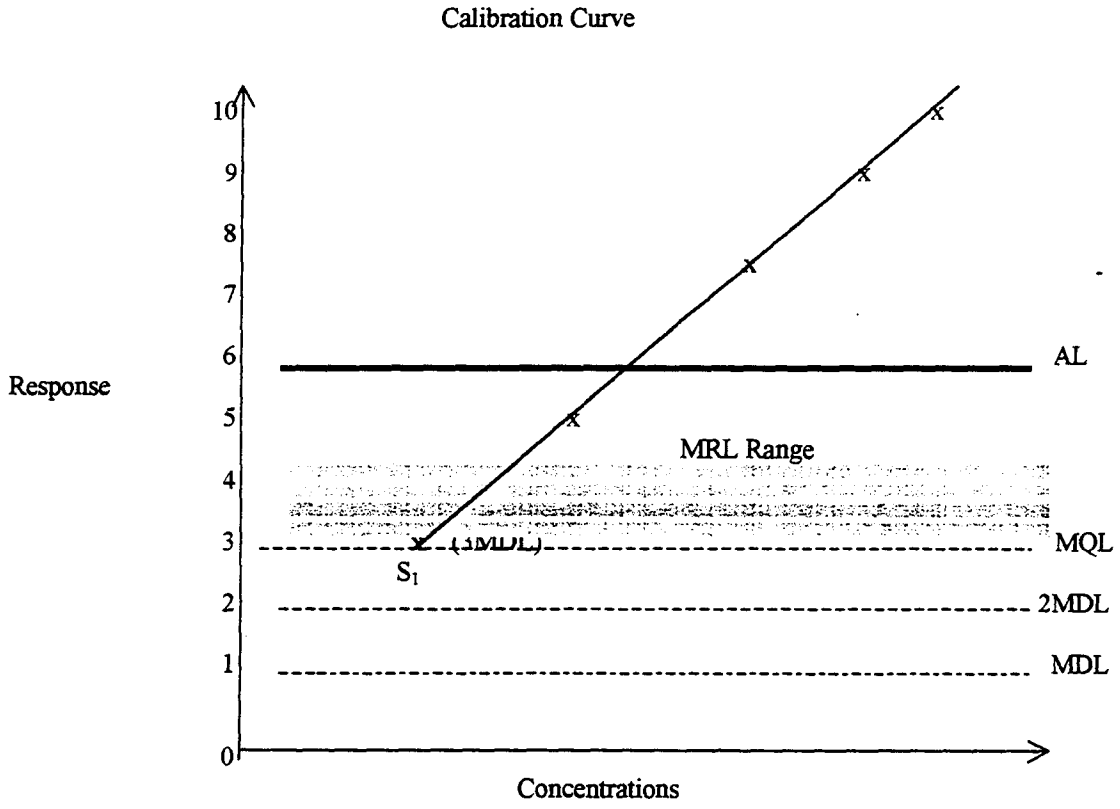
4. MRL: Method Reporting Limits are threshold values above which results are reported as positive quantities, and below which results are reported as non-detect (<) and flagged with "U", or as estimated and flagged with "J". MRL must not be established at levels below the MDL check sample level. MRL 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

Appendix F - Review Comments

Comment Responses

Document: **Quality Assurance Project Plan (QAPP), Limited Site Inspection for Ash Pits 1 & 3**
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 Plan submittals.
2.

Response: We apologize for not obtaining an OEPA, DERR preferred format to develop the Quality Assurance Project Plan (QAPP). The QAPP, FSP and DQO attachments were prepared for the Former Plum Brook Ordnance Works using the EPA Region 5 model QAPP. We apologize for this inconvenience. Future phases of work for this Formerly Used Defense Site, will incorporate the OEPA, DERR preferred format.

3. Section 1.2.1, Facility/Size and Borders: Please indicate the size and borders of Ash Pits 1 & 3.

Response: Both Ash Pits are approximately 1 acre in size. Ash pit 1 is located south of Maintenance Road and north of Powerhouse 1. The northern and southern boundaries of ash pit 1 are still somewhat defined by berms or rises in elevation. The northern and southern berms transverse in an east/west direction along Maintenance Road and Powerhouse 1 respectively. The eastern and western boundaries of the pit are not clearly defined by a berm or rise in elevation, but have been identified from historical aerial photographs and drawings. Ash pit 3 is located west of Ransom Road and south of Maintenance Road within the boundaries of the NASA K-site research facility (formerly Powerhouse 3). The eastern border of Ash Pit 3 is the K-site research facility building. The remaining borders to the north, south, and west are thick vegetation. This information will be added to Section 1.2.1.

4. General Comment: The Figures, Referenced in the text of the QAPP and the Field Sampling Plan, are not labeled.

Response: The Figures will be labeled and referenced accordingly.

5. Field Sampling Plan, Section 4.0, Sampling Design and Rationale, page 7: The first sentence of the first bullet, for Ash Pit 1, is missing the number 1 following the word "pit".

Response: The number 1 will be added to the text following the word "pit".

U.S. ARMY CORPS
OF ENGINEERS
LOUISVILLE DISTRICT

Field Sampling Plan

Limited Site Investigation of the former

Plum Brook Ordnance Works

Ash Pits Numbers 1 and 3

Sandusky, Ohio

DERP-FUDS HTRW Project No. G05OH001821

July 2000

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FIGURES

Figure 1 - Ash Pit 1

Figure 2 - Ash Pit 3

FIELD SAMPLING PLAN

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 the Department of Defense (DoD) as a manufacturing plant for trinitrotoluene (TNT), dinitrotoluene (DNT), and pentolite. Contamination detected at the site by several studies has been related to past activities and are being addressed under the Defense Environmental Restoration Program (DERP), Formerly Used Defense Sites (FUDS). A limited Site Investigation (SI) of two former ash pits located within PBOW will be conducted to evaluate the potential for contamination that may have resulted from past DoD activities.

This project is one of several limited SI projects at PBOW, 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 Installation

Land for the original site was acquired by Department of War in 1938 and consisted of approximately 9,010 acres of land. In the early 1940s the U.S. Army contracted with Trojan Powder Company to manufacture 2,4,6-TNT, dinitrotoluene and pentolite at PBOW. Production began on 16 December 1941 and continued through late 1945, ceasing two weeks after V-J Day (2 September 1945). After operations ceased, the area was turned over to Army Ordnance Department and renamed Plum Brook Depot and used for ammunition storage.

PBOW was placed in standby status from 1945 to 1946. During this time, decontamination and decommissioning (D&D) procedures were performed on many structures associated with the manufacturing process. D&D included the removal and relocation of all explosives to burn grounds for destruction by open burning. Where possible, remaining structures and buildings were burned in place. Drain lines and steam lines were flushed and dismantled.

In December 1945, custody of PBOW was transferred from Trojan Powder Company to U.S. Army Ordnance Department, with the U.S. Army Corps of Engineers assuming custodial responsibilities from 1 January through 30 June 1946. In August 1946, PBOW was transferred to the War Assets Administration.

In 1956 the National Advisory Committee for Aeronautics (NACA) began leasing sections of PBOW from the War Assets Administration. An agreement was made in 1956 to lease 500 acres in the northern portion of PBOW to construct and operate the Plum Brook Reactor Facility (PBRF). In 1958 NACA changed its name to National Aeronautics and Space Administration (NASA). By 1963, approximately 6,400 acres of PBOW had been acquired by NASA for various aerospace research activities. An additional 2,000 acres were acquired to serve as a buffer zone between the facility and the adjacent community. Research and test activities were conducted by NASA throughout the 1960s and have continued until today.

NASA declared approximately 2,150 acres as excess in 1978. Forty-six acres of the excessed property is used by the Perkins Township Board of Education as a bus transportation center. Much of the remaining excess property was reclaimed as farmland. NASA also excessed Parcel #59 to the General Services Administration (GSA) for subsequent disposal.

1.2.2 Site Specific

The focus of this site investigation will be the former ash pits 1 and 3. The locations of the subject ash pits are shown in Figures 1 and 2. During the period when PBOW was operational, DoD constructed and utilized three power stations corresponding to the Power and Maintenance areas 1, 2, and 3. Each power station consisted of a main powerhouse, a coal storage area, and a diked area containing fuel oil above ground storage tanks (ASTs). Each power house building consisted of a boiler house, compressor room, electrical room, filter room, and locker room. The buildings also contained two to four large coal-burning boilers, a turboelectric generator, a feed water treatment system, and several steam driven or electric air compressors. The generated steam was used for space heating, driving compressors, and generating electrical power. Coal ash generated from each of the boilers in the powerhouse was collected in pits. Water was added to the ash and the water/ash slurry flowed through a sluice trench to the ash sump located at the end of the building. From the ash sump, the ash traveled through a pipeline to a nearby surface water impoundment (ash pit) (D&M, 1997).

Information concerning the status and/or use of the ash pits following the war is limited. During the 1960's, the power plant facility at Power, and

Maintenance Area 3 was remodeled to become the K Site Test Facility (NASA-PBS) (MK, 1994).

1.3 Climate

The climate for Erie County is continental with cold and cloudy winters and warm 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 monthly rainfall average with 1.65 inches, whereas July had the highest monthly rainfall average 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 (MK1994: DM, 1997).

1.4 Topography

According to the United States Geological Survey (USGS) quadrangle 7.5, ash pit 1 is located primarily on a flat grassland area at an elevation of approximately 635 feet mean sea level (msl). The ash pit is located south of Maintenance Road and north of Powerhouse 1. Powerhouse 1 and associated railroad tracks are still present at the site. The former ash pit is now covered with a thicket of grass, weeds, and small trees. The northern and southern boundaries of the ash pit are still somewhat defined by berms or rises in elevation approximately 3 to 5 feet. The northern and southern berms transverse in an east/west direction along Maintenance Road and Powerhouse 1 respectively. Both of these berms are approximately 640 feet msl. The eastern and western boundaries of the pit are not clearly defined by a berm or rise in elevation, but have been identified from historical aerial photographs and drawings. A drainage ditch is located on the northern boundary of the ash pit and runs in a northerly direction and under Maintenance Road by means of a concrete culvert.

Ash pit 3 is primarily located on a flat grassland area at an elevation of approximately 635 feet msl. The ash pit is located west of Ransom Road and south of Maintenance Road. The Powerhouse 3 area has been reconfigured by NASA and is now a test facility (K-site). The surrounding area around the ash pit is at an elevation of 640 feet msl. The K-site test facility is currently supplying non-contact cooling water to the former ash pit, which is now holding water. The depth of the water in the pit has not been ascertained. A pipe at the northeastern corner of the pit is supplying the cooling water. It is assumed that this pipe was the original pipe that once supplied the ash to the pit. The boundaries of the pit appear to be the same as the original boundaries, with the exception of an indention to the west on the eastern boundary. Railroad tracks running in a north-south direction are still present at the site. The pit is partially surrounded by mature and smaller trees. The water is discharged from the former pit by means of

an east-west trending surface water ditch that eventually discharges into Pipe Creek.

1.5 Geology

Two formations of the Devonian Age underlay both ash pits 1 and 3. The oldest formation is the Delaware Limestone, which is characterized as a hard, dense, finely crystalline limestone and dolomite. Dissolution of this unit has produced solution channels along bedding planes and joints, and even producing caverns in some areas. The unit is typically buff colored and is fossiliferous. Overlaying the Delaware Limestone is the Olentangy Formation. This formation is made up of two members, the Plum Brook Shale and the overlying Prout Limestone. Only the Plum Brook Shale member should be encountered at the ash pits. 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 Olentangy formation is the first natural boundary beneath the ash pits at an approximate depth of 25 feet below ground surface. (IT, 1999)

The overburden soils at both ash pits is predominately composed of glacial till, outwash, or lacustrine (lake) deposits. It is assumed that the overburden at both pits is approximately 25 feet thick (IT, 1999). The surface soil at both sites is of the Kibbie-Elnora-Tuscola-Colwood Association (ODNR, 1994). This association is described as nearly level to gently sloping. The soil is described as somewhat poorly drained, moderately well drained, and very poorly drained formed in outwash, lucrastine, and deltaic sediments. Based on review of boring logs advanced in the vicinity of the former ash pits, overburden soils generally consists of an upper layer of silty fine sand and silt of low plasticity. These materials typically are classified as SM and ML, respectively, in accordance with the Unified Soil Classification System (USCS). This upper layer is generally underlain by silty clay of low to high plasticity extending to bedrock. This material was typically classified as CL or CH in accordance with the USCS. (IT,1999)

1.6 Hydrogeology

Based on a review of the site wide groundwater study, ground water is encountered in the bedrock units underlying the ash pit sites. Generally the groundwater flows northward toward Lake Erie. Groundwater in the overburden soils is generally expected to be perched or trapped water. Perched water occurs in irregular, discontinuous granular zones within the soil overburden. Perch water sources contain widely varying quantities of water depending on recent precipitation. Based on expected soil types, overburden groundwater quantities if encountered, should be minimal.

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 contracted the Louisville District (CELRL) to execute the design and fieldwork for the investigation. Several institutions and individuals will coordinate efforts to complete the investigation. Their names and functions are listed below.

2.2 Planning Team Members

PBS/NASA - Keith Peecook
Project Manager: Rick Meadows, CELRH-DL-M
Project Scientist: Darrell Davis, CELRL-ED-EB

2.3 Louisville Technical Team

Project Scientist: Darrell Davis, CELRL-ED-EB
Industrial Hygienist: Shelton Poole, CELRL-ED-EB
Safety Auditor: Shirley Dunn, CELRL-SO
Risk Assessor: David Brancato, CELRL-ED-EE
Hydrogeologist: Martin Wahking, CELRL-ED-EB
QA/QC Chemist: Samir Mansy, CELRL-ED-EB
Technical Team Leader: Doug Meadors, CELRL-ED-EE

3.0 SCOPE AND OBJECTIVES

The purpose of this limited SI is to evaluate the potential for contamination of the site due to past DOD 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, if warranted by the analytical results, will be performed in subsequent phases.

The Field investigation for this limited SI will include:

- A visual survey to assess and document site conditions
- Surface and Subsurface soil sampling
- Surface water and Sediment sampling

Target parameters include semivolatile organic compounds (SVOCs), and metals. 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).

Geotechnical and additional environmental data will be gathered to help assess the physical and potential migration characteristics of the subject sites. This includes pH, total organic carbon (TOC), soil grain size distribution, in accordance with ASTM D422, and soil plasticity (Atteberg Limits) in accordance with ASTM D4318.

4.0 SAMPLING DESIGN AND RATIONALE

The constituents of concern (COC) associated with coal ash, that may have been released to the environment include SVOCs and metals. The population to be represented by sampling in this investigation is the range of concentrations of COC that may exist in the surface water, sediment, surface and subsurface soil at Ash pits #1 and #3. Total Organic Carbon (TOC) analysis, pH, and geotechnical data will also be collected on soil samples to aid in assessing the potential for migration at the site. The potential release of contamination, the chemical properties of the suspected COC, the geological conditions, and potential transport mechanisms were utilized to support the recommended sampling strategy.

Populations of COC may exist in different soil strata, therefore, more than one stratum or matrix will be characterized by this sampling effort. Samples will be collected from the surface soil to characterize the population of COC in the area of the original release. Samples will also be collected from the subsurface soil to evaluate constituents that may be migrating downward through the overburden. Surface water and sediment samples will be collected to evaluate potential migration pathways associated with drainage. Runoff from ash pit 1 is to a drainage ditch to the north of the site. Surface water and sediment samples will be taken downstream to evaluate if COC have migrated via the drainage ditch. Ash pit 3 is now under water; therefore, only surface water and sediment samples will be obtained within the former pit. The east-west trending surface water ditch discharging water from the former ash pit 3 will also be evaluated by collecting surface water and sediment samples.

Ash pit 1 – Three hand auger borings will be advanced within the ash pit. Continuous sampling will be conducted in the borings. Two samples will be collected within each boring. One surface soil sample will be collected within the zero (0) to one (1) foot interval. The second sample will be collected within the three (3) to four (4) foot interval below ground surface (bgs). One set of surface water and sediment samples will be collected in the drainage ditch downstream and north of the ash pit. The approximate sample locations are illustrated in Figure 2. All samples will be analyzed for SVOCs, metals. In addition soil samples will be collected for TOC, pH, and geotechnical parameters as different soil strata are encountered. The locations for the samples are described below:

- The entry point of the ash into pit 1 will be sampled if it can be located. Based on professional judgement, this would be one of the most likely places to find it. This area of the pit would have been in direct contact with the ash and would have the greatest exposure time to COC.

- Two additional borings will be taken within the ash pit; however, their locations will be determined in the field by the field technical team leader. These borings will be advanced where there is visual ash residues. It has been stated that within the pit there are bare areas containing black gritty soil. It is assumed that these black gritty soils are the residual coal ash.

- Drainage and run off from ash pit 1 flows northward towards a drainage ditch. One set of surface water and sediment samples will be collected in this ditch downstream from the pit. A complete visual survey of the site will be performed to investigate any additional drainage, seeps or run-off to nearby soil or surface water. The decision to collect additional samples will be made in the field by the field technical team leader.

Ash pit 3 – This pit is currently under water. Three surface water and sediment samples will be collected for ash pit 3. These samples will be analyzed for SVOCs, and metals. The approximate sample locations are illustrated in Figure 3. The locations for the samples are described below:

- One surface water and sediment sample will be collected at the northeast corner of the pit. This location was selected because it is assumed that this is where the ash entered the pit. It is also where NASA periodically releases non-contact cooling water into the former ash pit. This area of the pit would have been in direct contact with the ash and would have the greatest exposure time to the COC.

- One surface water and sediment sample will be collected where the water leaves the pit via a drainage ditch.

- One surface water and sediment sample will be taken down gradient of the ash pit in the drainage ditch leading to a tributary of Pipe Creek.

Table 1 - Summary of Sampling Activities for the Site Investigation

Location	Field Effort	Sampling Activities	Chemical Analyses
Ash pit 1	• Visual Inspection • Surface and Subsurface Sampling	• Assess Site • Drill three borings (Hand Auger). One boring will be located where ash entered the pit. Two more borings will be located where visual ash residue is present. Continuous sampling will be performed. Samples will be collected at one foot (surface) and at 3-4 feet (subsurface) in the soil column.	• SVOCs • Metals • TOC • Geotechnical
	• Surface water and sediment sampling	• Collect surface water and sediment samples in the drainage ditch northwest and down gradient of ash pit.	• SVOCs • Metals
Ash pit 3	• Visual Inspection • Surface water and sediment Sampling	• Assess Site • Collect surface water and sediment sample at the northeastern corner where ash and water entered pit. • Collect surface water and sediment sample where ash and water exited the pit. • Collect surface water and sediment sample down gradient of ash pit in surface water drainage ditch.	• SVOCs • Metals

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 and will constitute master field investigation documents. Information to be recorded in the logs includes, but is not limited to the following:

- Project Identification.
- Subject of the field activity.
- General work activity, work dates, and general time of occurrence.
- Unusual events.
- Communication with the project manager and facility representative.
- Weather conditions (ambient air temperature, sky conditions, precipitation, and personal observations of wind conditions).
- Visitors on site.
- Sample number and time of day for each sample collected for analysis.
- Record of telephone calls to laboratory informing it of sample shipment.
- Accomplishment of decontamination of sampling equipment.
- Decontamination fluids, and soil cuttings.
- Variances from project plans and procedures.
- Accomplishment of tailgate safety meetings.
- Photographs taken and identification numbers (including location, spatial orientation, and a brief description of the photograph subject).
- Results of visual inspections.

5.3 Photographs

Color photographs will be taken of sampling areas to record significant field observations and to record site conditions at the time of the field investigation. All pictures will be time and date stamped and will be logged in the field logbook. 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 Surface and Subsurface Soil Sampling

Surface soil sampling will be performed using a stainless steel hand auger. The area around the boring location will be cleared of any debris (twigs, rocks, litter). A piece of plastic sheeting will be placed on the ground around the sample boring location to prevent cross contamination. Samples will not come into direct contact of this plastic sheeting. Hand augers will be decontaminated prior to being used. The boring will be advanced by turning the hand auger's crossbar at the same time the operator presses the auger into the ground. Sampling will be continuous throughout the boring. Samples will be collected at approximately 1 foot and within the 3-4 foot interval. If there are visual signs of contamination, the hand auger will be advanced an additional 2 to 3 feet and samples will be collected or composited. Any variances in sampling due to field conditions or findings will be properly documented. All borings will be documented with a hand auger boring log and soils will be visually classified using the Unified Soil Classification System (USCS). Once the proper depth is obtained the auger is removed from the borehole and the sample is taken from the sampling section of the auger. Samples will be placed in a stainless steel bowl and will be thoroughly mixed and homogenized before placing samples in the appropriate container. All soil sample containers will be clearly identified, and preserved on ice at 4 ° degrees Celsius. The proper volume of sample will be taken to insure that all internal laboratory quality control samples (i.e., Matrix Spike/Matrix Spike Duplicates (MS/MSDs), spikes, lab duplicates) can be performed by the laboratory. Sample collection information will be recorded in the field logbook. All sampling equipment will be appropriately decontaminated prior to each individual sampling episode to prevent down-hole and cross-hole contamination and prior to leaving the investigative area.

5.5 Surface Water and Sediment Sampling

Each surface water and sediment sample will be collected from the same location. The water sample will be collected first and sediments second to minimize collection of sediment within the water samples. The sampling sequence will begin at downstream locations and progress upstream to prevent cross-contamination from one location to another. Water samples will be collected by dip sampling. The sample container will be filled by holding the container just beneath the surface of the water. If access is limited, a clamp and pole will be used to extend the reach of the sampling team member. Water samples that need acid preservative will be preserved after the sample has been collected. Sediment samples will be collected by using a stainless steel scoop. If access is limited or the depth of sediment is too great a clamp and pole will be used to extend the reach of the sampling team member. Effort will be made to obtain sediment samples beneath the water sediment interface at the same location where the surface water was

collected. Any excess water obtained while collecting sediment samples will be decanted from the sediment. Both water and sediment samples will be collected facing upstream. All surface water samples requiring preservation will be preserved in the field after the samples are in the appropriate container. Sediment samples will be placed in a stainless steel bowl and homogenized, before placing samples in containers. All samples will be clearly identified on the sample labels and put on ice for preservation at 4 ° Celsius. The proper volume of sample will be taken to insure that all internal laboratory quality control samples (i.e., MS/MSDs, spikes, lab duplicates) can be performed by the laboratory. Sample collection information will be recorded in a field logbook. All sampling equipment will be decontaminated prior to each individual sampling episode to prevent cross contamination and prior to leaving the investigative area.

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 investigative samples. Duplicate samples will be used to monitor sampling precision in the field. Duplicate analysis will be performed for SVOC, and metals analyses.

- Field Blanks - Field Blank samples will be collected by obtaining a portion of the deionized water used for rinsing sampling equipment (i.e., hand augers, stainless steel scoops) during decontamination procedures. Field Blanks will be collected in the appropriate sample container. One Field Blank will be collected for every 20 investigative samples. These blanks will monitor if the deionized water being used in the field has introduced any contamination into the samples. SVOC, and metals analyses will be performed on field blanks.

- Temperature Blanks - These samples will be prepared by adding deionized water to a sample bottle. This sample 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 equipment during the sample collection process.

Personnel entering exclusion zones during field activities must decontaminate upon exiting the area. Personal safety and health considerations are presented in the Site-specific Health and Safety 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, sediment, and water 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 all suspect contaminated equipment down with brush scrubbing and the soap solution. Hand tools, trowels, scoops, bowls, bailers, etc. used for sample collection of soils, sediments, ground and surface waters shall similarly be decontaminated between samples and before leaving the site for the day. Decontamination fluids will require containerization approved for liquids, labeled and properly stored, while awaiting disposal. Based on the anticipated levels of contamination on most sites, USACE has obtained approval from NASA-PBS and Ohio Environmental Protection Agency

(OEPA) to dispose 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

A critical aspect of sample collection and analysis protocols is the maintenance of strict 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. All aspects of 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, COC to be analyzed, and to minimize breakage during transportation. Sample bottle size required, preservatives, and holding times are listed in the table below. 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
- project number
- collectors initials
- date and time of collection
- preservatives added
- sample type
- depth

Table 2 - Summary Table of Sample Containers, Preservation Methods, and Holding Times for Water Samples

Parameter	Analytical Method	Quantity	Type	Preservation Methods	Holding Times
SVOCs	8270	2	1-liter, narrow-mouth amber glass, Teflon®-lined cap	Cool, 4°C	Extraction: 7days Analysis: 40 days
Metals	6010/7417	1	1-liter, polyethylene bottle	HNO_3 to pH<2	6 months mercury - 28 days

Table 3 - Summary Table of Sample Containers, Preservation Methods, and Holding Times for Soils Samples

Parameter	Analytical Method	Quantity	Type	Preservation Methods	Holding Times
SVOCs	8270	1	4-ounce, wide-mouth, glass, Teflon®-lined cap,	Cool, 4°C	Extraction: 14days Analysis: 40 days
Metals	6010	1	4-ounce, wide-mouth, glass, Teflon®-lined cap,	Cool, 4°C	6 months mercury - 28 days
TOC & Geotechnical	Walkly-Black	1	4-ounce, wide-mouth, glass, Teflon®-lined cap,	Cool, 4°C	28 Days

6.2 Sample Identification

Each sample will be assigned a unique identification number that will identify each sample for laboratory analysis. The sampling will consist of 12 alphanumeric characters separated in 5 groups, as shown in the example and explained below.

PBOW 99 SS A1 05

Group 1 (5 characters or less): identifies the project
Group 2 (2 digits): identifies the year
Group 3 (2 characters): identifies the type of sample according to the following code:

SS	surface soil
SB	soil boring (subsurface soil)
SW	surface water
SD	sediment

Group 4 (2 characters): identifies the location of the sample according to a code particular for each site. For this project the codes are:

A1 - Ash pit 1
A3 - Ash pit 3

Group 5 (2 digits): sequential number of samples taken at the location.

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 temperature 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°C ± 2° 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. Soil cuttings from all hand-augured borings will be replaced for backfilling borings. All personal protective equipment (PPE) (e.g., Tyvek®, nitrile or latex gloves) will be placed in a plastic garbage bag and taken to a Plum Brook Station dumpster for disposal. All

decontamination water will be collected and stored in an appropriate storage container. After all sampling is completed the water will be placed in a sanitary sewer system at Plum Brook Station. USACE has obtained approval and concurrence from NASA-PBS and OEPA in the on site management of IDW.

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 #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 preparation.

- 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 QAPP and all attachments prior to the actual 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 sampling equipment, logbooks, sample containers, labels, chain of custody forms, shipping supplies, coolers, and packing materials, etc.

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). 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.

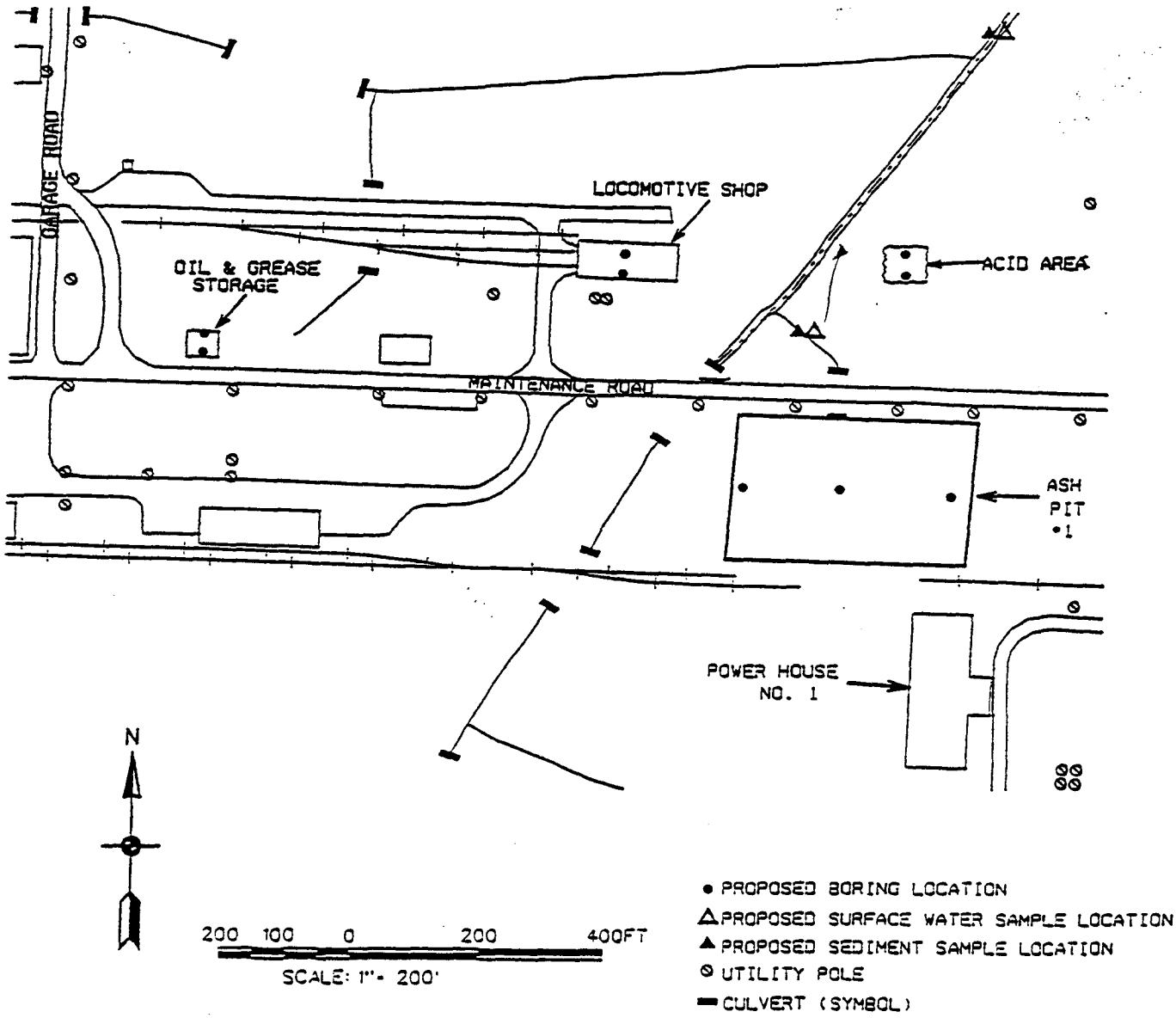

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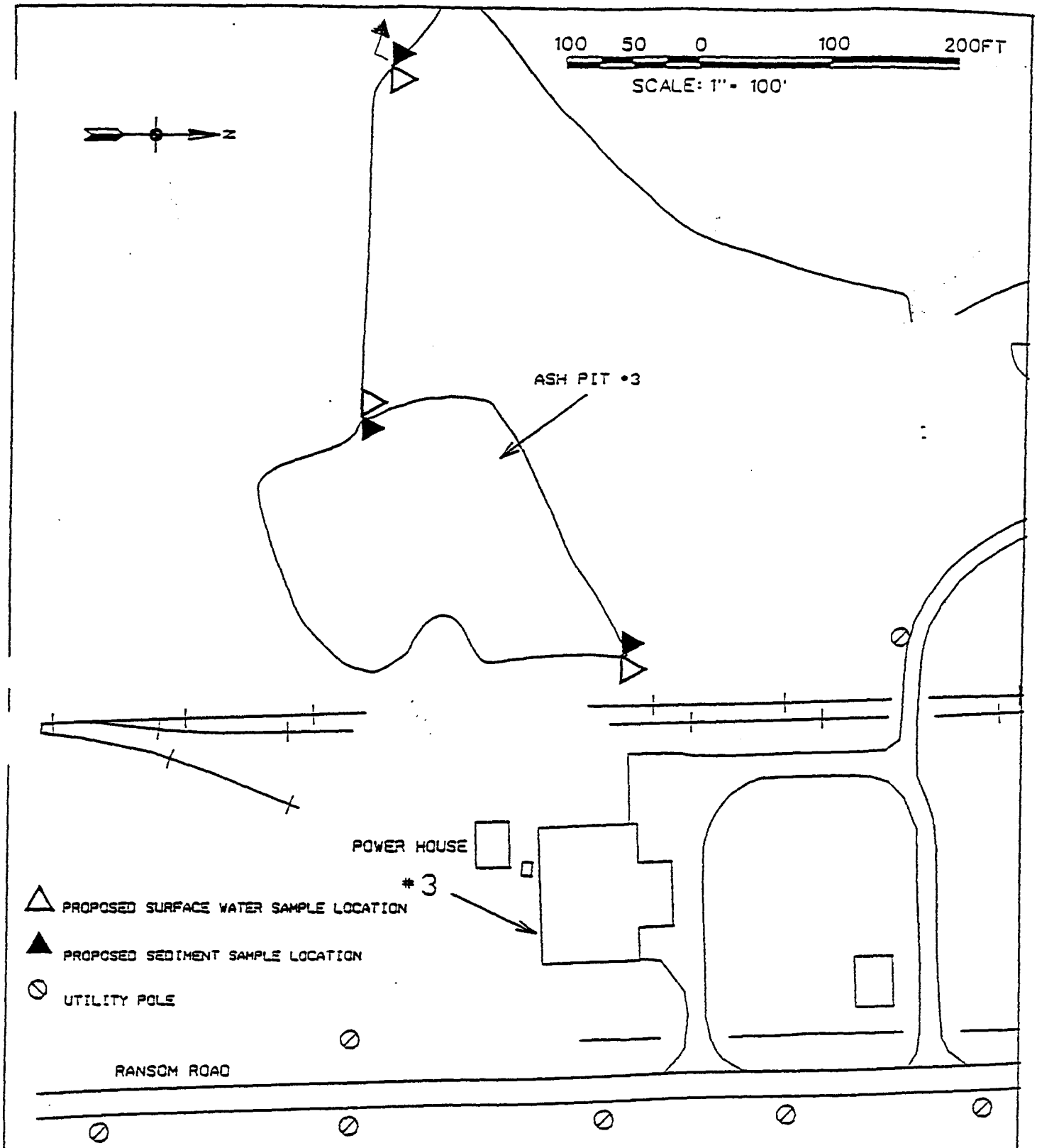
Figure #1



Design By:	Drawn By:	Checked By:	US Army Corps of Engineers	PLUM BROOK STATION SANDUSKY, OHIO LIMITED SITE INVESTIGATION FY 99	PROPOSED SAMPLE LOCATIONS ASH PIT #3
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Figure #2

U.S. ARMY CORPS
OF ENGINEERS
LOUISVILLE DISTRICT

Data Quality Objectives

Limited Site Investigation of the former

Plum Brook Ordnance Works

Ash Pits Numbers 1 and 3

Sandusky, Ohio

DERP-FUDS HTRW Project No. G05OH001821

July 2000

DQO Process: Limited Site Investigation
Plum Brook Station - Ash Pits #1 & #3

The purpose of this section is to provide and document the rationale for developing Data Quality Objectives (DQOs) for the Site Investigation (SI) of Ash Pits #1 and #3 located at the Plum Brook Station (PBS), Sandusky, Ohio.

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, where to collect samples, how many samples should be collected, and the tolerable level of decision errors for the study.

The DQO process consists of the following seven distinct steps with the following brief descriptions

- **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 Errors** – 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 any steps in the process and improve the investigation as knowledge of the site increases.

BACKGROUND

Ash Pit #1 is located south of Maintenance Road and west of Taylor Road. In March 1995, Dames & Moore conducted a field reconnaissance of the ash pit area north of Power Station #1. A depression was observed between the railroad tracks and Maintenance Road. The depression contained small trees, grasses, and some bare areas having black gritty soil. Drainage from this area is to a culvert under Maintenance Road. The location of this depression corresponds to the location of Ash Pit #1 in historical drawings. No surface water, sediment, surface soil, subsurface soil, or ground water samples have been taken at Ash Pit #1. A site wide ground water study was performed by Dames & Moore, 1997. Several semi-volatile organic compounds (SVOC) and metals were detected in the ground water study above their regulatory action levels; however, it could not be determined if Ash Pit #1 contributed to these detections.

Ash Pit # 3 is located at what is now NASA's site test facility, west of Ransom Road and south of Maintenance Road. During a field reconnaissance of this area in the fall of 1994, representatives of Dames & Moore and CELRN observed a surface water impoundment west of the NASA K-site building. Water flowed into the impoundment through a steel pipe at its northeast corner, and water discharged from impoundment into an east-west trending surface water ditch, which discharges into Pipe Creek. The impoundment corresponds to the location of Ash Pit #3 that appears in historical drawings. It was discovered that NASA is currently pumping non contact cooling water from its K-site test facility into the former Ash Pit #3. No surface water, sediment, surface soil, subsurface soil, or ground water samples have been taken at Ash Pit #3. NASA has a NPDES permit and test the cooling water on a monthly basis for pH, temperature, and flow rates. A site-wide ground water study was performed by IT Corp. in 1999. Several SVOCs and metals were detected in the ground water study above their regulatory action levels; however, it could not be determined if Ash Pit #3 contributed to these detections.

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.

Identification of Planning Team Members

OEPA – Ron Nabors
PBS/NASA - Keith Peecook
Project Manager: Rick Meadows, CELRH-DL-M
Project Scientist: Darrell Davis, CELRL-ED-EB

Louisville Technical Team:

Project Scientist: Darrell Davis, CELRL-ED-EB
Industrial Hygienist: Shelton Poole, CELRL-ED-EB
Safety Auditor: Shirley Dunn, CELRL-SO
Risk Assessor: David Brancato, CELRL-ED-EE
Hydrogeologist: Martin Wahking, CELRL-ED-EB
QA Manager/Chemist: Samir Mansy, CELRL-ED-EB
Technical Manager: Doug Meadors, CELRL-ED-EE

Description of Problem. The potential problem regarding Ash Pits #1 and #3 was identified in an Inventory Project Request (INPR) performed by Huntington District, as requesting a limited site investigation to evaluate the potential for surface and subsurface soil contamination. The storage of coal ash at the pits may have resulted in the release of constituents of concern (COC) into the soil. Based on the chemical properties of coal ash, COC include SVOCs and metals. No previous investigations have been performed at the Ash Pits. The approximate locations of the Ash Pits have been identified from historical drawings and aerial photographs of PBOW. No information is available detailing how much fly ash was stored within the Ash Pits. It must be determined if residual COC in the surface and subsurface soil at Ash Pits #1 and #3 are at concentrations that exceed the media-specific screening criteria. Detection of COC in the surface soil would confirm that a release occurred to the soil. Detections of the same COC in the subsurface soil would indicate the downward infiltration of COC has occurred under the influence of precipitation and other physical or chemical processes.

Transport Mechanisms. Mechanisms for the potential release of COC from the Ash Pits include:

- Downward migration through the soil column to subsurface soils and ground water.
- Run-off to nearby surface water and sediment.

Section 1.3.1 describes the site/installation geology, hydrology, and hydrogeology. The general lithologic conditions at PBS are characterized as glacial till. Sands, silt, and clays make up most of the overburden soil layer. There are some pockets of perched water or sand lenses containing water present within the overburden. Shale and limestone bedrock are the first natural boundaries beneath the site at an approximate depth of 25 feet below ground surface. The physical and chemical properties of the native soils at the site as well as the physical and chemical properties of the COC will influence transport mechanisms. Ground water will not be investigated as part of this limited SI. CELRN and IT Corp. are currently addressing site wide ground water.

Resources and Relevant Deadlines for the Site Investigation.
The funding resources for SI are provided under the DERP-FUDS DoD program on a Quarterly basis for FY99. The SI must be completed by the end of FY99. Louisville District Environmental Engineering Branch will provide the necessary technical expertise and resources to the investigation based on the investigation's technical requirements and schedule.

Step 2: Identify the Decision: The purpose of this step is to define the decision statement that the study will attempt to resolve.

Principal Study Question. Do COC exist in the environmental media at Ash Pits #1 and #3 at levels that exceed USEPA Region 9 Preliminary Remediation Goals (PRGs)?

Alternative Actions that Could Result from Resolution of the Principal 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 investigation and/or interim action.

Decision Statement. The primary decision for the site investigation is to determine whether COC are present at Ash Pits #1 and #3 at levels that exceed media specific screening criteria (Region 9 PRGs) and thus would require further action.

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.

Informational Required to Resolve Decision Statement.

- Historical records, interviews, aerial photographs, visual inspections, previous environmental investigations, site topography, geology, site hydrology and hydrogeology will be evaluated. This information will be utilized to make an informed decision about the expected type of COC, as well as, the most likely locations where contaminated media will be discovered.

- Transport mechanisms and chemical properties of COC to evaluate migration pathways. It should be noted that development of a Conceptual Site Model is beyond the scope of this limited SI.

- Analytical samples and results from the corresponding environmental media within the Ash Pit areas to compare to screening criteria.

Sources for information Identified Above. DERP-FUDS guidance in conjunction with EPA guidances are the principal tools guiding the investigation. CELRH has an extensive administrative record (AR) for the former PBOW. A review of this AR and other resources revealed useful information about historical records, interviews, aerial photographs, visual inspections, previous environmental investigations, site topography, geology, hydrology and hydrogeology. Information regarding the chemical properties and characteristics of COCs once identified will be obtained and included in the final SI report discussion and recommendations.

Information Needed to Establish the Action Level and Confirm that Appropriate Measurement Methods Exist to Provide the Necessary Data. The action levels for the COC will be the USEPA Region 9 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. These performance-based methods have inherited quantitative and qualitative QA objectives, internal method requirements, and specific QC limits. These methods along with strict USACE QA/QC guidance and protocols will provide data that will meet data quality objectives.

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.

Characteristics that Define the Population of Interest. The COC associated with fly ash that may have been released to the environment include SVOCs and metals. The population to be represented by sampling in this investigation is the range of concentrations of COC that may exist in the soil, water, and sediment at Ash Pits #1 and #3. The potential release of contamination, the chemical properties of the suspected COC, the geological conditions, and transport mechanisms will support the need for the recommended sampling strategy.

Populations of COC may exist in different soil strata, therefore, more than one stratum should be characterized by sampling. Samples should be collected from the surface soil to characterize the population of COC in the area of the original release. Samples will also be collected from the subsurface soil to evaluate constituents that may be migrating downward through the overburden. Runoff from ash pit #1 is to a drainage ditch to the northwest of the site. Surface water and sediment samples will be taken downstream to evaluate if COC have migrated via the drainage ditch. Ash Pit #3 is now under water; therefore, only surface water and sediment samples will be obtained within the former pit. The east-west trending surface water ditch discharging water from the pit will also be evaluated by taking surface water and sediment samples.
These samples will be used to evaluate if COC have migrated via this ditch.

Spatial Boundary of the Decision Statement. The site investigation will focus on the storage area of fly ash identified in historical drawings, aerial photos, and site visits. The approximate size of the area to be investigated at Ash Pit #1 is 2 acres. The approximate size of the area being investigated at Ash Pit #3 is 1 acre. The horizontal extents of the site investigation boundaries are delineated in **Figures 1 and 2.** The vertical extent of the site investigation is expected to be approximately 4 feet below ground surface for Ash Pit #1, and the sediment for Ash Pit #3. A visual survey of the surrounding areas around the Ash Pits will be performed in the field to investigate drainage, run off, and seeps. Should field observations indicate COC exist at deeper depths, hand auger extensions will be available access deeper soils.

Temporal Boundary of Decision Statement. The analytical data obtained from this site investigation will be used as valid indicators of COC throughout an exposure timeframe of 50 years. The sampling for this site investigation would be best performed during the early spring through fall seasons. This would allow for favorable weather conditions during fieldwork.

Scale of Decision Making. The scale of decision making will be concentrations of the SVOCs and metals in the first 4 feet of the overburden in Ash Pit #1, and the concentrations of COC in the surface water, and sediment at both Ash Pits, that exceed media-specific screening criteria. The analytical data obtained from the sampling effort will play a crucial role in any and all decisions, and recommendations made in regards to ash pits #1 and #3.

Practical Constraints on Data Collection. Potential constraints on the site investigation could vary from scheduling problems, sampling procedures, weather, and budget constraints.

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.

Specify the Parameter that Characterizes the Population of Interest. The maximum concentrations of COC detected in the analytical samples will be compared to specific screening criteria. Any values exceeding these criteria will be the main target of interest.

Specify the Action Level for the Site Investigation. The analytical sample results will be compared to the appropriate media-specific screening criteria from Region 9 PRGs. The levels of the screening criteria are intentionally conservative so that protection of human health and the environment may be achieved.

Decision Rule. If levels of COC exceed the screening criteria (PRGs), recommendations for further investigation and/or interim action will be developed. If COC do not exceed screening criteria, OEPA will be contacted to proceed towards a No Further Action (NFA) decision document. It should be noted that if multiple elements or compounds are encountered in the media, $1/10^{th}$ of individual PRG values will be utilized as the screening criteria.

This limited SI includes minimal samples that will be obtained from locations most likely contaminated (biased). Therefore, each sample result will be compared to screening criteria and statistical evaluations will be minimal.

Step 6: Specify Tolerable Limits on Decision Errors:

The purpose of this step is to specify the decision makers tolerable limits on decision errors which are used to establish performance goals for the data collection design.

Determine the Possible Range of the Parameter of Interest. No previous environmental samples have been collected at the Ash Pits. 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.

Identify the Decision Errors and Choose the null Hypothesis. A non-statistical sampling design will be employed to select locations for sampling and determine the number of samples required. The non-statistical approach has been selected due to the limited nature of this SI and the lack of environmental data. This sampling design will be purposeful and biased towards locations most likely to be contaminated. Four steps are used to define where each decision error occurs relative to an action level and established the decision errors associated with the sampling design. This process is described below:

Definition of Decision Errors. As in any statistical hypothesis test, there are two kinds of errors that can occur in implementing the decision rule: the hypothesis may be rejected when true (Type I error, with probability α), or accepted 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 COC 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 condition or null hypothesis (H_o) for the ash pits is "COC detected in soil are at concentrations that warrant additional investigation activities". The alternate hypothesis (H_a) is "COC detected in soil are at concentrations that do not warrant additional investigation activities". In terms of this investigation, the default assumption (null hypothesis) is that the concentrations of COC at the site are significant enough to require further investigation.

Potential Consequence of each Decision Error. The consequence of deciding that the suspected COC detected in the soil do not exceed appropriate screening criteria when they actually do (i.e., Type I error) is the possible endangerment of human health and the environment. In this situation, the U.S. Army could be liable for damages encountered in the future and the associated environmental cleanup costs. Alternatively, the consequence of deciding that the COC in soil exceed appropriate screening criteria when they truly do not (i.e., Type II error) is that the U.S. Army would incur unnecessary expenses associated with additional investigative 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 is likely to be most significant in terms of the stated null hypotheses. The Type I decision error has more severe consequences since the risk of jeopardizing human health and the environment is likely to far outweigh the consequences associated with additional investigation expenditures. Because of the limited nature of this SI and lack of existing data, enough data will not be collected to confidently assure a Type I error will not be encountered. This SI will serve to get a baseline condition of the site. Therefore, to assure protection of human health and the environmental, it is understood that additional investigation will most likely be recommended and implemented to verify that a Type I error does not exist.

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. The site investigation will be based on surface and subsurface soil, surface water, and sediment samples collected within the ash pits. As more information is obtained, the sampling design will be optimized to accomplish the goals of the site investigation.

Develop a General Sampling and Analysis Design. No existing environmental data are available for Ash Pits #1 and #3. 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. The following is the rationale for the proposed location and number of samples:

Ash Pit #1 – Three hand auger borings will be drilled within the ash pit. Continuous sampling will be conducted in the borings. Two samples will be collected within each boring. One surface soil sample will be collected 0-1 ft. The second sample will be collected in the range of 3-4 ft. bgs. The selection of these depths will best represent the various environmental media that need to be investigated. These depths will also confirm if there has been some downward migration through the soil column of the COC. The soil samples will be analyzed for SVOCs, metals. Geotechnical samples will be collected dependent upon the soil's cohesiveness. The decision to take geotechnical samples will be determined in the field. One set of surface water and sediment samples will be collected in the drainage ditch downstream and north west of the ash pit.The location of the borings can be seen in **Figure 1**. The rationale for the sampling design and locations are listed below:

1. If possible the entry point of where the ash was delivered to the pit will be identified. If contamination is present, this would be one of the most likely places to find it. This area of the pit would have been in direct contact with the ash and would have the greatest exposure time to COC.

2. Two additional borings will be taken within the ash pit; however, their locations will be determined in the field. These borings will be taken where there is visual ash residues. It has been stated that within the pit there are bare areas containing black gritty soil. It is assumed that these black gritty soils are the residual ash.

3. Drainage run off from Ash Pit #1 flows westward towards a drainage ditch. One set of surface water and sediment samples will be collected upstream from the pit, in this ditch. A complete visual survey will be performed of the site to investigate any additional drainage, seeps or run-off at the site to nearby soil or surface water. The decision to collect additional samples will be made in the field.

Ash Pit #3 – This pit is currently under water. Four surface water and sediment samples will be collected for Ash Pit #3. The sampling locations are illustrated in Figure 2. These samples will be analyzed for SVOCs, and metals. The rationale for the sampling design and locations are listed below:

1. One surface water and sediment sample will be collected at the northeast corner of the pit. This location was selected because it is where the ash entered the pit. It is also where NASA is presently releasing non-contact cooling water into the former ash pit. This area of the pit would have been in direct contact with the ash and would have the greatest exposure time to COC.

2. One surface water and sediment sample will be collected where the water leaves the pit via a surface water drainage ditch.

3. One surface water and sediment sample will be taken down gradient of the ash pit in a tributary of Pipe Creek.