U.S. ARMY CORPS
OF ENGINEERS
LOUISVILLE DISTRICT

Quality Assurance Project Plan

Limited Site Investigation of the former

Plum Brook Ordnance Works

Lower Toluene Tank Area

Sandusky, Ohio

DERP-FUDS HTRW Project No. G05OH001824

July 2000

200-1e

G05OH001824_01.14_1001_a

TABLE OF CONTENTS

TITLE PAGE

TABLE OF CONTENTS

LIST OF ACRONYMS

1.0 PROJECT DESCRIPTION

1.1 Introduction

1.2 Site Description

1.2.1 Facility / Site Size and Borders
1.2.2 Topography
1.2.3 Local Geography & Hydrogeology

1.3 Past Data Collection Activities

1.4 Current Status

1.5 Project Objectives

1.5.1 Project Target Parameters and Intended Data Usages
1.5.2 Quality Objectives and Criteria or Measurement Data

1.6 Sample Rationale

1.6.1 Sample Network by Task and Matrix
1.6.2 Site Maps of Sampling Locations
1.6.3 Rationale of Selected Sampling Locations

1.7 Project Schedule

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 Project Organization Chart
2.2 Management Responsibilities
2.3 Quality Assurance (QA) Responsibilities
2.4 Field Responsibilities
2.5 Laboratory Responsibilities

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

3.1 Precision

3.1.1 Definition
3.1.2 Field Precision Objectives
3.1.3 Laboratory Precision Objectives

3.2 Accuracy

3.2.1 Definition
3.2.2 Field Accuracy Objectives
3.2.3 Laboratory Accuracy Objectives

3.3 Completeness

3.3.1 Definition
3.3.2 Field Accuracy Objectives
3.3.3 Laboratory Completeness Objectives

3.4 Representativeness

3.4.1 Definition
3.4.2 Measures to Ensure Representativeness of Field Data
3.4.3 Measures to Ensure Representativeness of Laboratory Data

3.5 Comparability

3.5.1 Definition
3.5.2 Measures to Ensure Comparability of Field Data
3.5.3 Measures to Ensure Comparability of Laboratory Data

3.6 Level of Quality Control Effort

4.0 SAMPLING PROCEDURES

5.0 CUSTODY PROCEDURES

5.1 Field Custody Procedures
5.2 Laboratory Custody Procedures
5.3 Final Evidence Files

6.0 CALIBRATION PROCEDURES AND FREQUENCY

6.1 Field Instrument Calibration
6.2 Laboratory Instrument Calibration

7.0 ANALYTICAL PROCEDURES

7.1 Field Analytical Procedures
7.2 Laboratory Analytical Procedures
7.3 List of Project Target Compounds and Laboratory Detection Limits

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Field Quality Control Checks
8.2 Laboratory Quality Control Checks

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

9.1 Data Reduction
9.1.1 Field Data Reduction Procedures
9.1.2 Laboratory Data Reduction Procedures

9.2 Data Validation
9.2.1 Procedures Used to Evaluate Field Data
9.2.2 Procedures Used to Validate Laboratory Data

9.3 Data Reporting

9.3.1 Field Data Reporting
9.3.2 Laboratory Data Reporting

10.0 PERFORMANCE AND SYSTEM AUDITS

10.1 Laboratory Performance and System Audits

10.1.1 Internal Laboratory Audits
10.1.1.1 Internal Laboratory Audit Responsibilities
10.1.1.2 Internal Laboratory Audit Frequency
10.1.1.3 Internal Laboratory Audit Procedures

10.1.2 External Laboratory Audits

10.1.2.1 External Laboratory Audit Responsibilities
10.1.2.2 External Lab Audit Frequency
10.1.2.3 Overview of the External Laboratory Audit Process

11.0 PREVENTATIVE MAINTENANCE

11.1 Field Instrument Preventative Maintenance
11.2 Laboratory Instrument Preventative Maintenance

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

12.1 Accuracy Assessment
12.2 Precision Assessment
12.3 Completeness Assessment

13.0 CORRECTIVE ACTION

13.1 Field Corrective Action
13.2 Laboratory Corrective Action
13.3 Corrective Action During Data Validation and Data Assessment

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.1 Contents of Project QA Reports
14.2 Frequency of QA Reports
14.3 Individuals Receiving / Reviewing QA Reports

REFERENCES

FIGURES

Figure 1: Vicinity Location Plan
Figure 2: Site Location Plan
Figure 3: Sample Location Plan
Figure 4: MK MW15 Boring Log
Figure 5: MK MW14 Boring Log

APPENDICES

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

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

AST	Above Ground Storage Tank
BTEX	benzene, toluene, ethylbenzene, xylene
bgs	below ground surface
CELRH	Corps of Engineers, Great Lakes and Ohio River Division, Huntington District
CELRL	Corps of Engineers, Great Lakes and Ohio River Division, Louisville District
CERCLA	Comprehensive Environmental Response, Compensation, Liability Act of 1980
CERCLIS	Comprehensive Environmental Response, Compensation, and Liability Information System
COC	chain of custody
D&D	decontamination & decommissioning
DERP	Defense Environmental Restoration Program
DNT	dinitrotoluene
DOD	U.S. Department of Defense
ERIIS	Environmental Risk Information & Imaging Services
ERNS	Emergency Response Notification System
FUDS	Formerly Used Defense Sites
gpm	gallons per minute
GSA	General Services Administration
HTRW	Hazardous, Toxic, and Radiological Waste
HWS	Ohio Master Site List

IDW	Investigation-Derived Waste
INPR	Inventory Project Request
IT	IT Corporation
LeRC	Lewis Research Center
LRST	Ohio Leaking Underground Storage Tank Report
MSDS	Material Safety Data Sheet
msl	mean sea level
NACA	National Advisory Committee for Aeronautics
NASA	National Aeronautical and Space Administration
NFA	No Further Action
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
PID	Photoionization Detector
ppb	parts per billion
PRGs	Preliminary Remediation Goals
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control

RCRIS_CA	Resource Conservation and Recovery Information System- TSD's Subject to Corrective Action
RCRIS_LG	Resource Conservation and Recovery Information System- Large Quantity Generators
RCRIS_SG	Resource Conservation and Recovery Information System- Small Quantity Generators
RCRIS_TS	Resource Conservation and Recovery Information System- NON-Corrective Action TSD Facility
RST	Ohio Underground Storage Tank Report
SAP	sampling and analysis plan
SI	site investigation
SWF	Ohio Solid Waste Facility List
TAL	target analyte list
TOC	Total Organic Carbon
TNT	trinitrotoluene
V-J Day	Victory over Japan day (September 2, 1945)
USACE	U.S. Army Corp of Engineers
USCS	Unified Soil Classification System
USEPA	U.S. Environmental Protection Agency

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 the lower toluene tank area 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 lower toluene tank area was identified in an Inventory Project Request (INPR) (Huntington District U.S. Army Corps of Engineers (USACE), 1998). A limited Site Investigation (SI) of the tank area 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.

During the period that PBOW was operational, DoD constructed and utilized six storage tanks that served as bulk storage for toluene used in the production of TNT. PBOW had three TNT production areas A, B, and C. Each production area was supported by two aboveground storage tanks (AST) used for bulk toluene storage. Each tank was approximately 30 feet in diameter, 41 feet high, constructed of steel, and had a capacity of 200,000 gallons. Each tank rested on a concrete foundation surrounded by a 1-foot wide by 1-foot high containment berm. The berm emptied into a 3-foot square drain valve pit. The pump for each tank is located at ground level near the drain valve pit. Historical photographs show much larger earthern berms around each tank which were approximately 55 feet from the center of the tank. More recent historical photographs do not show an earthern berm around the tanks. It has not been ascertained if these berms were removed by DoD, NASA, or naturally eroded away. The "Lower Toluene Tanks" supported "TNT Area C" during PBOW operations. The tanks were numbered 645 (northern tank) and 655 (southern tank). The tanks were installed in 1941, and were in service until 1945 and are illustrated in Figure 3. Toluene was transported to and from the tank area by railroad. The tanks were decommissioned in 1945 by pumping out their contents, draining transfer lines, and opening top and bottom flanges for ventilation. NASA removed tank 655 from the lower tank area and installed it within the Plum Brook reactor facility.

1.2.1 Facility/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"

The Lower Toluene Tank Area is approximately 4 acres. It is located in the southwestern portion of PBOW. The Tank Area is bordered to the south by the intersection of Patrol road and Taylor Road. The area is bordered to the east by abandoned railroad tracks running north and south. The remaining borders to the north and west are thick vegetative and forested areas (Fig.2 & 3).

1.2.2 Topography

According to the United States Geological Survey (USGS) quadrangle 7.5 the lower toluene tank area is located primarily on a flat grassland area at an elevation of approximately 680 feet mean sea level (msl). The tank area is located north of the Taylor Road and Patrol Road intersection, and is southeast of "TNT Area C". Mature trees and vegetation surround the remaining tank. There are no buildings

within the area. Some abandoned single-track railroad lines are located to the east of the tank area. Precipitation runoff from the tank area accumulates in a nearby surface depression or pond west of the site, or flows northward to a tributary of Pipe Creek by means of a drainage ditch. The surface depression was identified in an Inventory Project Request (INPR) prepared by CELRH (USACE, 1998) for the lower toluene tank area, however it could not be clearly identified from historical or recent aerial photographs.

1.2.3 Geography & Hydrogeology

It is assumed that two formations of the Devonian Age underlay the lower toluene tank area. The lowermost and oldest formation is the Delaware Limestone. It is characterized as a hard, dense, finely crystalline limestone and dolomite. Dissolution of this unit has been described which has produced solution channels along bedding planes and joints, and even producing caverns in some areas. The unit is typically buff colored and usually is described as 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 tank area.. 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 tank area at an approximate depth of 10 feet below ground surface (IT, 1999).

The overburden at the tank area is predominately composed of glacial till, outwash, or lacustrine (lake) deposits. The overburden at the lower tank area is approximately 10 feet thick (IT, 1999). Based on a review of the site-wide groundwater study (IT, 1999), ground water is encountered in the overburden soils underlying the tank area. Groundwater in the overburden soils is generally expected to be perched or trapped water related to the permeable interface between the overburden and underlying bedrock. The groundwater encountered at this site generally occurs in the granular zones within the soil overburden. These sources of water contain widely varying quantities of water depending on recent precipitation. Based on expected soil types, overburden groundwater quantities, if encountered, should be minimal and unrelated to deeper potable ground water sources within the bedrock layer.

1.3 Past Data Collection Activities

Morrison Knudsen installed two monitoring wells, MK-MW-14 (installed north of removed Tank 655) and MK-MW-15 (installed north of Tank 645). The boring logs of these two wells (Figure 4 & 5) described the lithology of the overburden unit as brown silty sand with traces of clay and some organics, until bedrock is encountered at depths of 11.5 feet (MK-MW-14) and 9 feet (MK-MW-15). Toluene was not detected in any of the soil samples during the installation of these wells. Toluene has not been detected in groundwater samples collected from these wells.

1.4 Current Status

The remaining tank was decommissioned in 1945. The tank area has become over grown with thick vegetation and trees.

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.
- Surface water and sediment sampling.

1.5.1 Project Target Parameters and Intended Data Usages

The target parameter for this investigation is toluene. Benzene, Toluene, Ethylbenzene, Xylenes (BTEX) analysis will be performed for these parameters and 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.

1.6 Sample Network Design and Rationale

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

1.6.1 Site Maps of Sampling Locations

Maps showing intended soil, sediment and surface water sampling locations are included in figure 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 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 asked the Louisville District (CELRL) to execute the design, fieldwork and technical reporting. Several institutions and individuals will coordinate efforts to carry out the project. Their names and functions are listed below:

Industrial Hygieniest:	Shelton Poole, CELRL-ED-EB
Project Scientist:	Darrell Davis, CELRL-ED-EB
Safety QC:	Shirley Dunn, CELRL-SO
Toxicologist:	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 Toxicologist

Dr. Brancato is well experienced in toxicology and 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 the Field Sampling Plan attached to this document.

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

The field instrument will be calibrated as described by the manufacturer's procedures. The field instrument used for this investigation is a photoionization detector (PID). As a rule, the calibration of the PID will be checked daily and recalibrated when required. Detailed information on the field screening is located in the Field Sampling Plan attached to this document.

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

The standardization and QA information for field measurements are described in Section 3 of this QAPP. Detailed information on the field screening is located in the Field Sampling plan attached to this document.

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 appendix E of this QAPP. Method detection limits shown above have been experimentally determined using the procedure found in 40CFR, Part 136, Appendix B, or equivalent statistical approach. The latest MDLs at the time of sample analysis will be used.

SECTION 8

INTERNAL QUALITY CONTROL CHECKS

8.1 Field Quality Control Checks

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

Detailed information on the field screening is located in the Field Sampling Plan attached to this document.

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:

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

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.

References

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

Dames & Moore, Inc., April 1997, *Records Review Report, Plum Brook Ordnance Works, Sandusky, Ohio.*

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

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

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

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

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

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

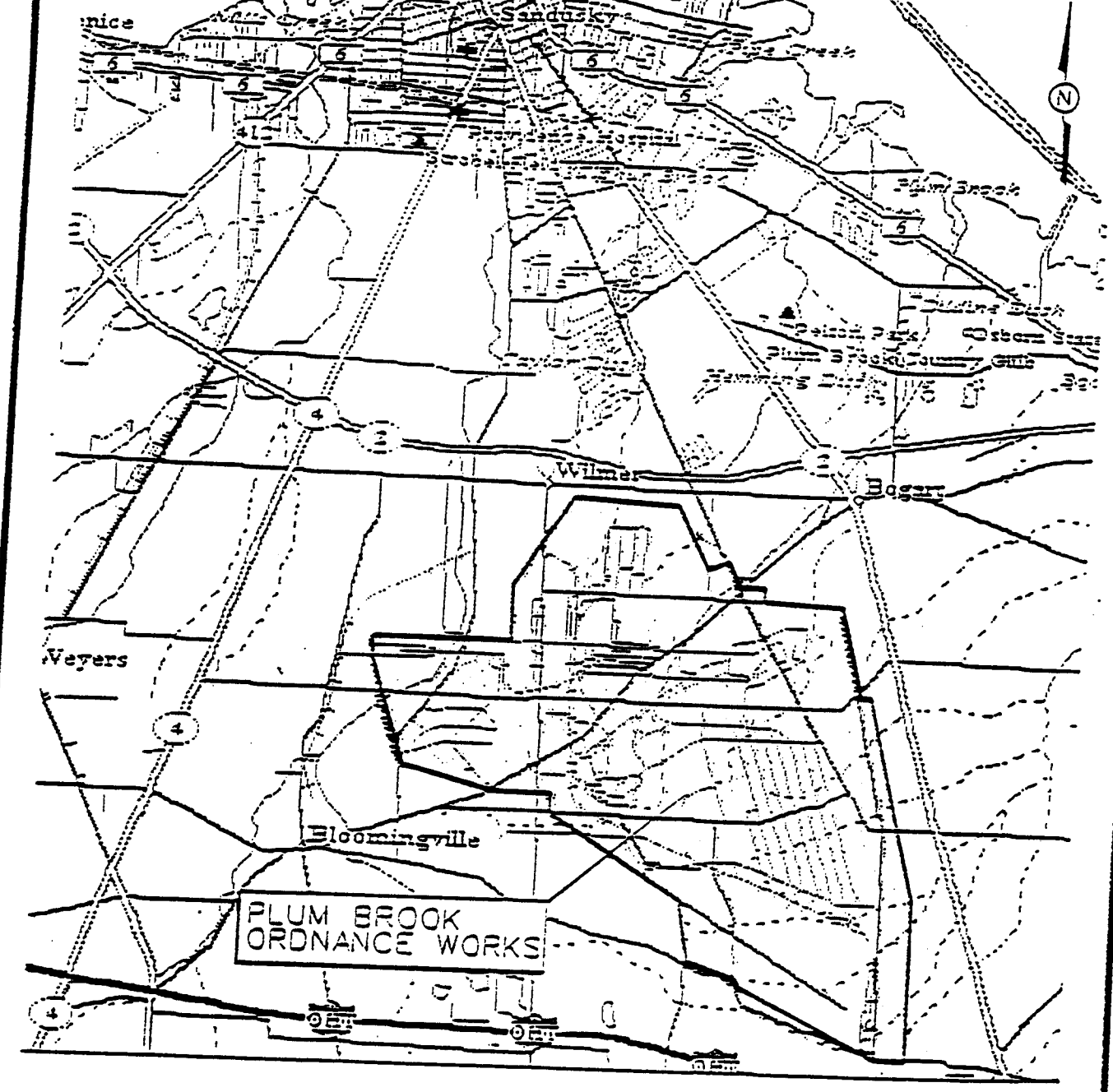
Morrison Knudsen Corporation (MK), 1994, *Site Inspection Report, Plum Brook Station, Sandusky, Ohio, Volume I,* (prepared for NASA), January.

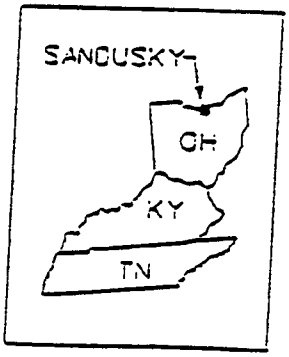
Plum Brook Ordnance Works (PBOW), *Archives Search Report,* USACE St. Louis District.

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

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

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

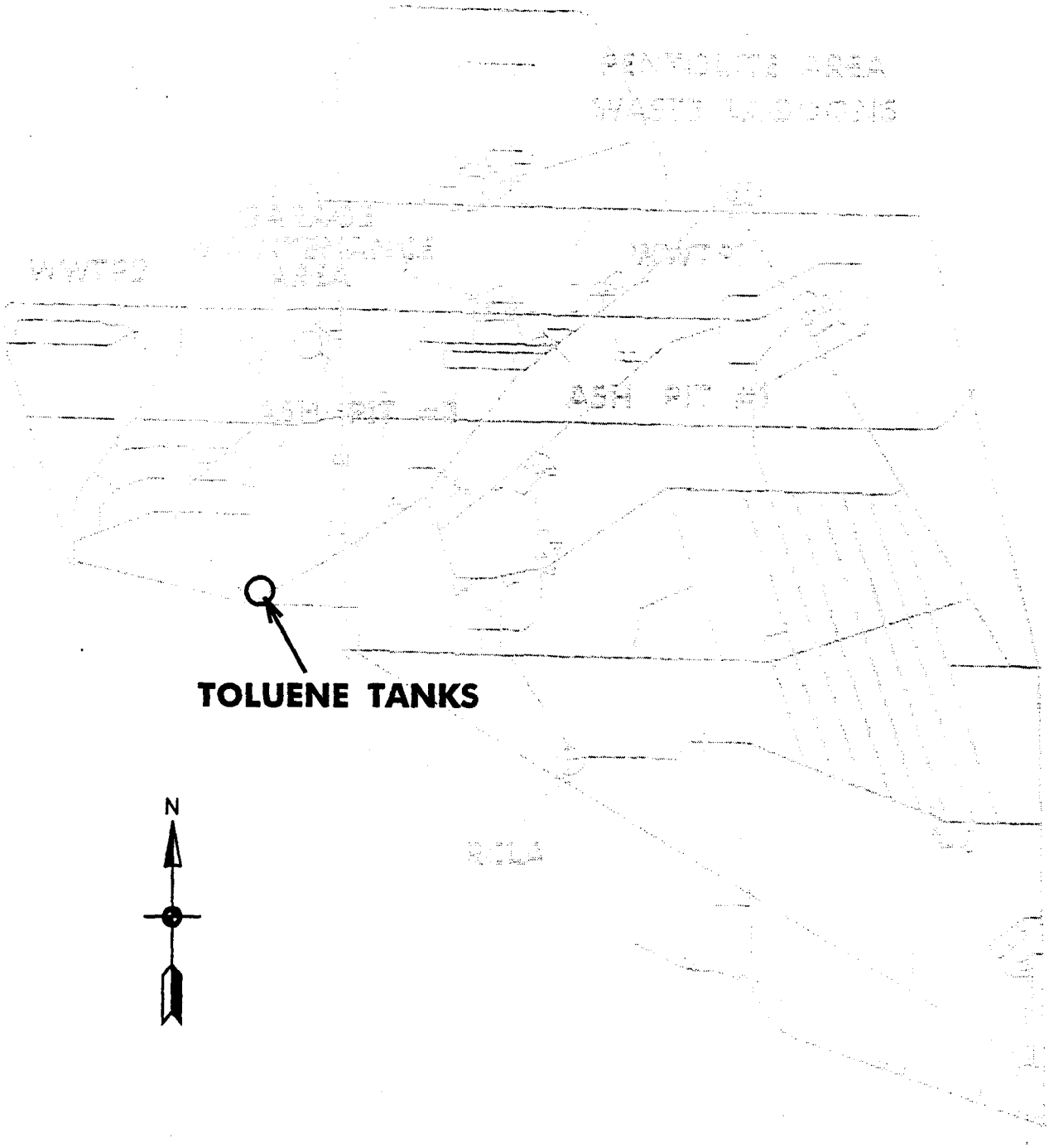




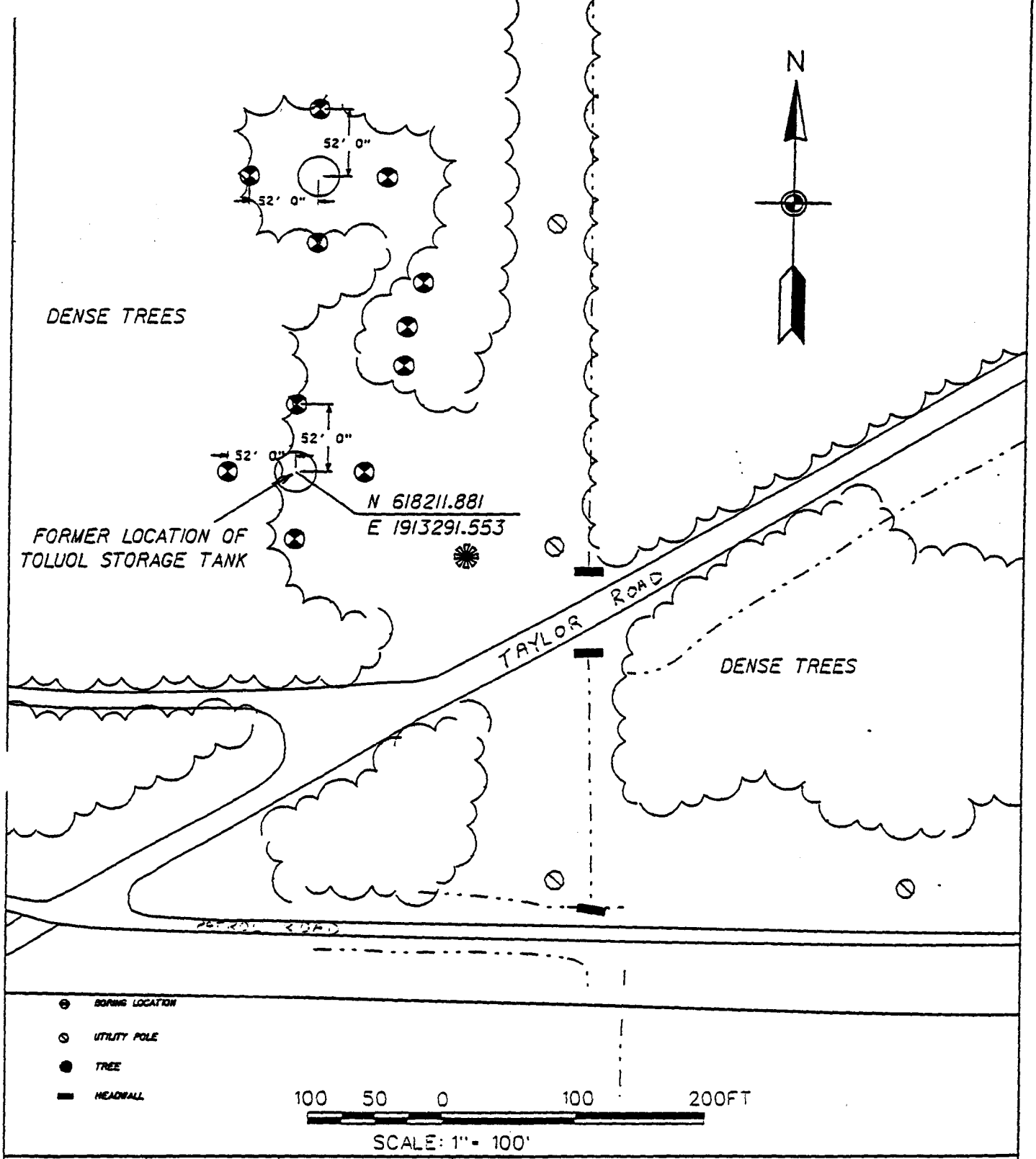
NOT TO SCALE

PLUM BROOK ORDNANCE WORKS
SANDUSKY, OHIO

FIGURE 1



Design By:	Drawn By: WDB	Checked By:	US Army Corps of Engineers	SITE LOCATION PLAN TOLUENE TANKS PLUM BROOK ORDNANCE WORKS SANDUSKY, OHIO	
Reviewed By:	Approved By:				
Date: AUG 1999	Scale: NONE		Drawing Code:	Sheet Ref. No.	FIGURE 2



Design By:	Drawn By:	Checked By:	US Army Corps of Engineers	PLUM BROOK STATION SANDUSKY, OHIO LIMITED SITE INVESTIGATION FY 99	PROPOSED SAMPLE LOCATIONS LOWER TOLUENE TANKS
Reviewed By:	Approved By:				
Date: 10 MAY 99	Scale: 1" = 100'	Drawing Code:	Sheet Ref. No.		FIGURE 3

File Name:
Plot Date:

MORRISON KNUDSEN CORP.
ENVIRONMENTAL SERVICES GROUP

SOIL BOREHOLE LOG

SHEET 1 OF 1

BOREHOLE NUMBER: MW15

PROJECT: PLUM BROOK SI	LOCATION: SANDUSKY, OHIO
EXCAVATION AUTHORIZATION NO.:	DRILLING SUB-CON: TOLTEST / RIG MODEL: TRACK
DATE START: 7/01/93 / FINISHED: 7/01/93	GEOLOGIST: T. BENSON / TOTAL DEPTH: 9'
ELEVATION: / COORDINATES:	BORING METHOD AND EQUIPMENT: HOLLOW STEM AUGER/SPLIT SPOON

GROUNDWATER LEVEL	DEPTH BELOW SURFACE (FEET)	SAMPLE: WELL SCREEN	METHOD	RECOVERY (INCHES)	RETAINED (INCHES)	OVA/HNU SCREENING ( )	SPT 6" -6" -6" -6"	N	USCS CLASIFICATION	SOIL DESCRIPTION: name, gradation, percent gravel, color, moisture content, relative density or consistency, soil structure, mineralogy, staining.
							2-3-4-5			0-9' SILTY SAND, BROWN, DRY TO SLIGHTLY MOIST, TRACE CLAY, SOME ORGANICS
▽										4' ENCOUNTERED WATER
	5									
										AUGERED TO 9', AUGER REFUSAL
										BEDROCK AT 9'
	10									TERMINATION @ 9'
	15									
	20									

BOREHOLE WATER LEVELS (NON-STATIC CONDITIONS)

WHILE SAMPLING: ft.	CASING DEPTH: ft.	WHILE DRILLING: ft.	CASING DEPTH: ft.
BEFORE REMOVING CASING: ft.	TOTAL CASING DEPTH: ft.	IMMEDIATELY AFTER REMOVING CASING: ft.	
AFTER COMPLETION: ft. DAY/TIME		AFTER COMPLETION: ft. DAY/TIME	

SOIL-LOG 3/26/93

FIGURE 4

MORRISON KNUDSEN CORP.
ENVIRONMENTAL SERVICES GROUP

SOIL BOREHOLE LOG

SHEET 1 OF 1

BOREHOLE NUMBER: MW14

PROJECT: PLUM BROOK SI	LOCATION: SANDUSKY, OHIO
EXCAVATION AUTHORIZATION NO.:	DRILLING SUB-CON: TOLTEST / RIG MODEL: TRACK
DATE START: 7/01/93 / FINISHED: 7/01/93	GEOLOGIST: T. BENSON / TOTAL DEPTH: 11.5'
ELEVATION: / COORDINATES:	BORING METHOD AND EQUIPMENT: HOLLOW STEM AUGER/SPLIT SPOON

GROUNDWATER LEVEL	DEPTH BELOW SURFACE (FEET)	SAMPLE: WELL SCREEN	METHOD	RECOVERY (INCHES)	RETAINED (INCHES)	OVA/HNU SCREENING ( )	SPT 6" -6" -6" -6"	N	USCS CLASIFICATION	SOIL DESCRIPTION: name, gradation, percent gravel, color, moisture content, relative density or consistency, soil structure, mineralogy, staining.
							2-2-2-3			0-11.5' SILTY SAND, NO GRAVEL, BROWN, MOIST, TRACE CLAY, SOME ORGANICS
	5									
▽										6' ENCOUNTERED WATER
	10									AUGERED TO 11.5' ENCOUNTERED BEDROCK @ 11.5', AUGER REFUSAL
										TERMINATION @ 11.5'
	15									
	20									

BOREHOLE WATER LEVELS (NON-STATIC CONDITIONS)

WHILE SAMPLING: ft.	CASING DEPTH: ft.	WHILE DRILLING: ft.	CASING DEPTH: ft.
BEFORE REMOVING CASING: ft.	TOTAL CASING DEPTH: ft.	IMMEDIATELY AFTER REMOVING CASING: ft.	
AFTER COMPLETION: ft. DAY/TIME		AFTER COMPLETION: ft. DAY/TIME	

FIGURE 5

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.

Lois Ezzo

(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;

o. 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;

o. 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 Rack *(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 Rack

(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, 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.

Alesia Mi Danford

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

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

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

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

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

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

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

(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 Bei *(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 Bei

(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.

Jon W. Rittenour

(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)

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. Ley

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)

QUANTERRA · ORTH 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 Geis, 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
  - Scott Irwin, Analyst
- General Chemistry, Open
  - Tenesha Roberts, Analyst
  - Dave Cox, Analyst
  - Julie Kuhle, Analyst (P)
  - Paulette Otto, Analyst
  - Kumudu Kulasekere, Analyst
  - Laurel Wood, Analyst
  - Sue Macenzak, Analyst
  - Melissa Fuller-Gustavel, Analyst
  - Tracey Dechiara, Dishwasher (P)

Report Production
Brad Belding

- Patty McClain, PA

Quality Assurance*
Opal Davis-Johnson

- Dorothy Leeson, QA Scientist
- Brenda Kravetz, QA Chemist
- Kristine Crock, Train Coordinator(P)

Finance
Lisa Long

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

*QA is included in North Canton headcount, but reports to Corporate.

GL - Group Leader
Data Rev - Data Entry
Pkg Gen - Package Generation Specialist

March 31, 1999

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIVING

(SUPERSEDES: REVISION 3, 04/18/97)

Prepared by: 6-4-97

Reviewed by: 6-4-97
Technology Specialist

Approved by: 6/4/97
Quality Assurance Manager

Approved by: 6-5-97
Environmental Health and Safety Coordinator

Approved by: 6-4-97
Laboratory Director

Proprietary Information Statement:

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

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

TABLE OF CONTENTS

1. PURPOSE *3*
2. RESPONSIBILITIES *3*
3. SAFETY *3*
4. PROCEDURES *4*
5. DEFINITIONS *10*
6. APPENDICES *10*

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. $^{6-}$	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	G	None	100 mL	TON	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

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B, 8021B, 8081A, 8082 and 8151A, SW-846

(SUPERSEDES: Revision 4)

Prepared by: Richard Burrows

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Health and Safety

Approved by: _____
Management

Proprietary Information Statement:

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

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

TABLE OF CONTENTS

1. Scope and Application 4
2. Summary of Method 4
3. Definitions 4
4. Interferences 4
5. Safety 4
6. Equipment and Supplies 5
7. Reagents and Standards 5
8. Sample Preservation and Storage 6
9. Quality Control 6
10. Calibration and Standardization 11
11. Procedure 16
12. Data Analysis and Calculations 19
13. Method Performance 23
14. Pollution Prevention 24
15. Waste Management 24
16. References 24
17. Miscellaneous 24

List of Appendices:

Appendix A — Analysis of Volatile Organics based on Methods 8010B, 8020A, and 8021A

Appendix B — Analysis of Organochlorine Pesticides and PCBs based on Method 8080A

Appendix C — Analysis of Phenoxy Acid Herbicides based on Method 8150B

Appendix D — Analysis of Organochlorine Pesticides and PCBs based on Method 8081

List of Tables

Table A1 Standard analyte list for 8010B, 8020A and 8021A

Table A2 Recommended conditions for method 8020A

Table A3 Recommended conditions for method 8010B

Table A4 Recommended conditions for method 8021A

Table A5 Surrogate and Internal standard concentrations for aqueous and low level soil samples, methods 8010B, 8020A, and 8021A

Table A6 Surrogate and internal standard concentrations for medium level soil samples, methods 8010B, 8020A and 8021A

Table A7 Concentrations for LCS and MS/MSD compounds, low level soil and aqueous, methods 8010B, 8020A and 8021A.

Table A8 Concentrations for LCS and MS/MSD compounds, medium level soil, methods 8010B, 8020A and 8021A

Table A9 Continuing calibration and initial demonstration limits, method 8010B

Table A10 Continuing calibration and initial demonstration limits, method 8020A

Table B1 Standard analyte and reporting limits, method 8080A

Table B2 Recommended conditions, method 8080A

Table B3 Calibration levels, method 8080A

Table B4 Column degradation evaluation mix, method 8080A

Table B5 LCS/matrix spike and surrogate levels, method 8080A

Table B6 LCS/matrix spike and surrogate levels for Aroclor analysis, method 8080A

Table B7 LCS/matrix spike and surrogate levels for TCLP, method 8080A

Table B8 Suggested analytical sequence, method 8080A

Table B9 Performance limits, method 8080A

Table C1 Standard analyte list, method 8150B

Table C2 Instrumental conditions, method 8150B

Table C3 Calibration standards, method 8150B

Table C4 LCS/Matrix spike and surrogate levels, method 8150B

Table C5 Performance limits, method 8150B

1. SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices.

2. SUMMARY OF METHOD

In general, semivolatile analytes are prepared for analysis using continuous or separatory funnel liquid / liquid extraction (SOP # CORP-OP-0001) or sonication or soxhlet extraction (SOP # CORP-OP-0001). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. In addition, some purge and trap autosamplers are susceptible to port specific contamination. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates. The following requirements must be met:

Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned

immediately. Refer to the Quanterra Chemical Hygiene plan for a complete description of personal protection equipment.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. Specific hazards are covered in the appendices.

5.1.1. Opened containers of neat standards will be handled in a fume hood.

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for gases are stored at -10 to -20°C. Other volatile and semivolatile stock standard solutions are stored at 4°C ±2°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Semivolatile stock standard solutions must be replaced after one year. (6 months if also used for 600 series methods). Stock standards of gases must be replaced at least every 2 months. (Every week if also used for 600 series methods). Other volatile stock standards must be replaced at least every 6 months (1 month if used for 600 series analysis) or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.1. Expiration times for all standards are measured from the time the standard is prepared, or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule.

7.2. Calibration Standards

7.2.1 Volatile Calibration Standards

The procedure for preparation of volatile standards is given in Appendix A

7.2.2. Semivolatile Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at 4°C ±2°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Argon/Methane.

7.4. Quality control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

Semivolatile extracts must be refrigerated at 4°C ±2°C and analyzed within 40 days of the start of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same

sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

9.2.1. Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery ±3 standard deviations for surrogates and LCS, and mean recovery ±2 standard deviations for matrix spikes. Precision limits for matrix spikes / matrix spike duplicates are zero to mean relative percent difference + 2 standard deviations.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable

- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If dual column analysis is used, the rules for which column's result to report are the same as for samples (Section 12.1). That is, the lower of the two results is reported.

9.4.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

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

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatiles samples, and sodium sulfate for semivolatiles soils tests (Refer to SOP No. CORP-OP-0001 for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of 9.5 mL of methanol as described in section 11.5 of Appendix A. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

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

9.6. Instrument Blanks

9.6.1. An instrument blank must be analysed with any sequence that does not contain a method blank.

9.6.2. A new sequence starts with any new initial calibration and if there has been a break of greater than 12 hours in sample analysis.

9.6.3. An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.6.4. Control criteria are the same as for the method blank, except that only reanalysis of affected samples would be required, not re-extraction.

9.7. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch; however, if the matrix spike and matrix spike duplicate are within limits, the batch may be acceptable.

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

9.7.2. If dual column analysis is used, the rules for which column's result to report are the same as for samples (Section 12.1). That is, the lower of the two results is reported.

9.7.3. LCS compound lists are included in the appendices.

9.7.4. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include 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.

9.8.1. If dual column analysis is used, the rules for which column's result to report are the same as for samples (Section 12.1). That is, the lower of the two results is reported.

9.9. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns and any changes in instrument operating parameters, including gas flows, detector temperatures, oven temperatures, etc.

10.2. With the exception of 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve.

10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

Equation 1

$$\text{Calibration Factor (CF)} = \frac{\text{Area or Height of Peak}}{\text{Mass Injected (ng)}}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. Use of peak area or height must be consistent. It is not permitted to switch between using peak area and height for quantitation within an analytical sequence.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If there is interference, the external standard approach must be used. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

Equation 2

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard
C_s = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Linear regression is a special case of the quadratic fit and may be used if the calibration range is sufficiently narrow so that a straight line will fit the calibration points. Average response factor may be used if the % RSD of the response factors or calibration factors is < 20%.

10.6.1. Average response factor

The average response factor may be used if the percent relative standard deviation (%RSD) of the response factors is < 20%.
The equation for average response factor is:

Equation 3

$$\text{Average response factor} = \frac{\sum RF_{1-n}}{n}$$

Where: n = Number of calibration levels

$\sum RF_{1-n}$ = Sum of response factors for each calibration level

10.6.2. Linear regression

The linear fit uses the following functions:

10.6.2.1. External Standard

Equation 4

$$Concentration = A + BR$$

Where: A = Intercept
B = Slope
R = Response

10.6.2.2. Internal Standard

Equation 5

$$Concentration = A + B\frac{(R \times C_{is})}{R_{is}}$$

Where the variables are defined in equations 2 and 4

10.6.3. Quadratic curve

The quadratic curve uses the following functions:

10.6.3.1. External standard

Equation 6

$$Concentration = A + BR + CR^2$$

10.6.3.2. Internal Standard

Equation 7

$$Concentration = A + B\left(\frac{R \times C_{is}}{R_{is}}\right) + C\left(\frac{R \times C_{is}}{R_{is}}\right)^2$$

Where: C = Curvature

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard deviation (%RSD) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard deviation is calculated as follows:

Equation 8

$$\%\ RSD = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N - P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

Note that when average response factors are used, this equation gives the same value as the %RSD of the response factors.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than the reporting limit for the analyte.
- Relative standard deviation of the calibration points from the curve used must be < 20%.

- Some data systems will not measure the %RSD from a linear or quadratic fit. In this case, the correlation coefficient may be used as an alternative to the %RSD, and must be greater than 0.995.

10.8.1. Weighting of data points

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

10.9. Calibration Verification

10.9.1. Continuing Calibration

The working calibration curve or RF must be verified by the analysis of a mid point continuing calibration standard at the beginning, after every 10 samples, and at the end of the analysis sequence (QC and instrument blanks included).

10.9.2. Daily Calibration

At least every 24 hours a daily calibration must be analyzed. The requirements of the daily calibration are the same as the continuing calibration with the addition that retention times are updated.

10.9.3. Any analyte that is reportable as found must have a % difference of $\leq$ 15% in the preceding continuing calibration, on the column used for quantitation. For dual column analysis, the column used for quantitation will be the column with the lower result. Methods 8010B and 8020A have different continuing calibration limits that are obtained from Table 3 of the reference method and are listed in Appendix A of this SOP.

10.9.4. For any analyte that is not reportable as found, the % difference may be -15% to -30%.

10.9.5. Reportable as found is defined as any analyte that would be reported as anything other than a non-detect.

10.9.6. If dual column analysis is used, at least one column must meet the criteria listed above. The other column must be within ± 30% difference from the initial calibration.

10.9.7. It is not necessary to run a continuing calibration standard at the beginning of the sequence if the first 10 samples are analyzed immediately after the completion of the initial calibration.

10.9.8. The last sample in the sequence must be followed by an ending calibration. The ending calibration serves the analyst in judging the validity of the sequence.

10.9.9. Methods 8010B and 8020A have different continuing calibration criteria. Criteria for continuing calibrations for these methods can be found in the appendices.

10.9.10. % Difference calculation

Equation 9

$$\% \text{ Difference} = \frac{\textit{Calculated}\text{ value} - \textit{Expected}\text{ value}}{\text{Expected value}} \times 100\%$$

10.9.11. Corrective Actions for Continuing Calibration

If the % difference for any analyte is > +30 to -15% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the response for any analyte still varies by more than 30%, a new calibration curve must be prepared.

10.9.12. Corrective Action for Samples

Any samples injected *after* the standard exceeding the continuing calibration criteria must be reinjected.

11. PROCEDURE

11.1. Extraction

Extraction procedures are referenced in the appendices.

11.2. Cleanup

Cleanup procedures are referenced in the appendices.

11.3. Gas Chromatography

Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

In general, volatiles analytes are introduced using purge and trap as described in Appendix A. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

11.5.1. The daily calibration includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.2. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multiresponse analytes (e.g., Aroclors) use the retention time of one major peak. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with

the mid point of the initial calibration and each daily calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than +/- 0.05 minutes, use +/- 0.05 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

11.6.5.1. The retention times of all compounds in each continuing calibration must be within the retention time windows established by the daily calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

- The retention time of that compound in the standard must be within a retention time range equal to twice the original window.
- No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

The center of the retention time windows determined in section 11.6 are adjusted to the retention time of each analyte as determined in the daily calibration standards. (See the method 8080A appendix for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each daily calibration, but not for the continuing calibration standards.

11.8. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP CORP-OP-0001 for determination of percent moisture.

11.9. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and / or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flags required). For confirmed results, the lower of the two results is reported.

12.1.2. If the % difference between the response on the two columns is greater than 50%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified.

12.1.3. Multi-response Analytes (Aroclors)

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.4. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the peaks in the level 3 standard, then the sample should be reanalyzed at a more concentrated dilution.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Samples

If internal standard calibration is used, then the internal standard response must be within 50 to 200% of the response in the preceding continuing calibration standard.

12.6. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.7. External Standard Calculations

12.7.1. Aqueous Samples

Equation 10

$$\text{Concentration } (\mu g / L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng, Section 10.1

12.7.2. Non-aqueous Samples

Equation 11

$$\text{Concentration } (\mu g / kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\text{Moisture}}{100} \qquad (D = 1 \text{ if wet weight is required})$$

A_x, V_t, D_f, CF and V_i are as defined in Equation 10

12.8. Internal Standard Calculations

12.8.1. Aqueous Samples

Equation 12

$$\text{Concentration } (\mu g / L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte
Ax, Df, Vs are as defined in Equation 10

12.8.2. Non-aqueous Samples

Equation 13

$$\text{Concentration } (\mu g / kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

All variables are as defined in equations 11 and 12

12.9. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

Equation 14

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Test Methods for Evaluating Solid Waste. Physical/Chemical Methods, SW846. 3rd Edition. Final Update 1, July 1992. Section 8000A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Section 7.6.8 of Method 8000A in SW-846 recommends reanalysis if the continuing calibration does not meet criteria and "if the initial analysis indicated the presence of specific target analytes that exceeded the criterion." This SOP is more rigorous than the requirements in SW-846 in that reanalysis is required if the continuing calibration exceeds - 15 to + 30% difference. whether or not the initial analysis indicated the presence of specific analytes.

17.1.2. Method 8000A in SW-846 recommends that the continuing calibration be within 15% difference from the calibration curve for all analytes. This SOP allows for a % Difference of + 30% and -15% for analytes that are not detected. This is supported by the statement in Method 8000A that reanalysis is only required "if the initial analysis indicated the presence of specific target analytes that exceeded the criterion." In any event. the % difference of the continuing calibration must be $\leq$ 15% for any analyte that is to be quantitated and reported.

17.1.3. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are

allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.1.4. Retention time windows are calculated as in SW-846. However, if a retention time window of less than 0.05 min. is calculated, the window defaults to 0.05 min.

17.1.5. Retention time windows are updated each day using the daily standard, as required by method 8000A. The system is not recalibrated unless the continuing calibration standards fall outside the retention time windows set by the daily standard.

17.2. Modifications from Previous Revision

17.2.1. Directions for methods 8081 and 8151 have been added.

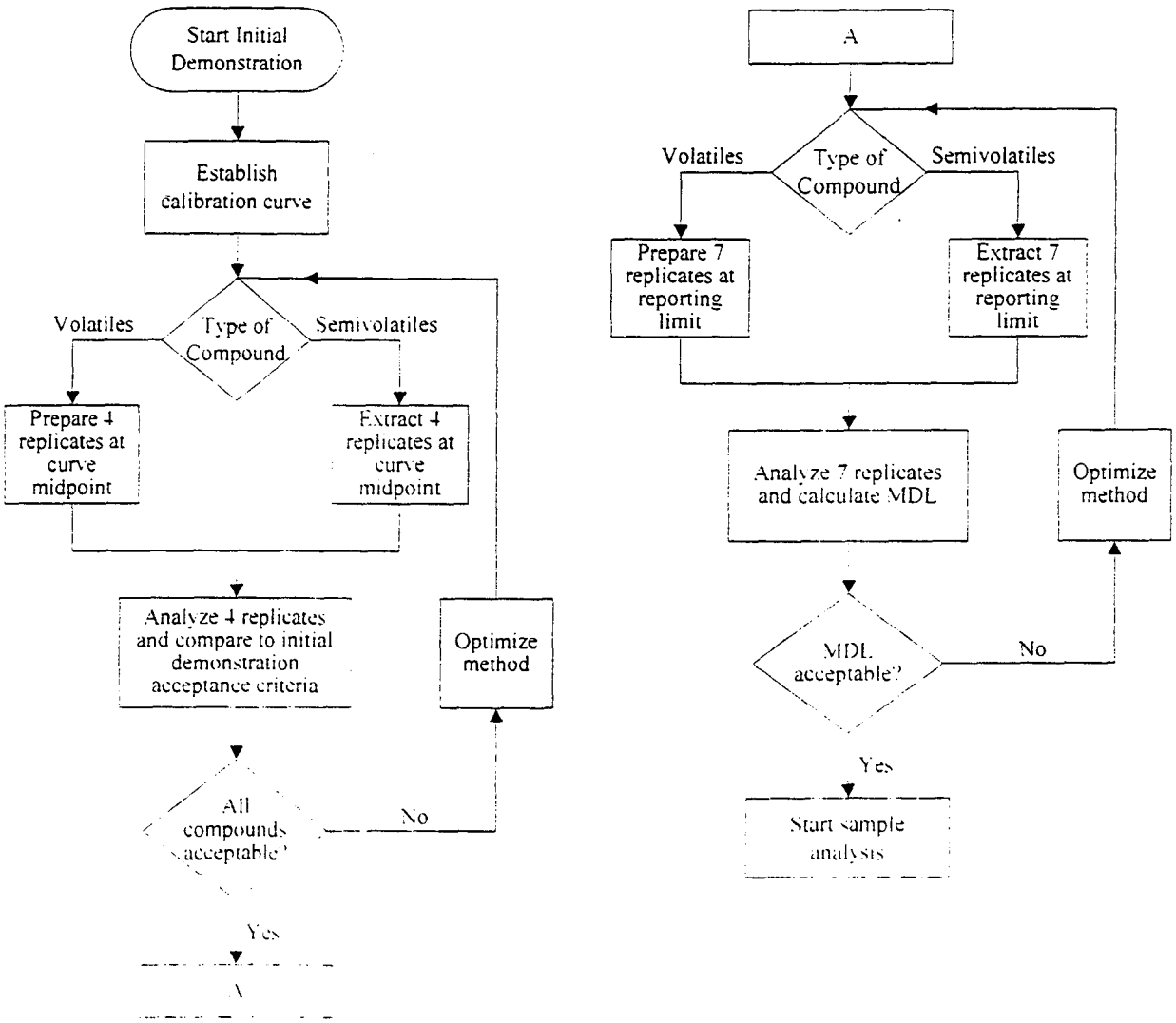
17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.3.1. Refer to the SOP change form on file in North Canton's Quality Assurance office.

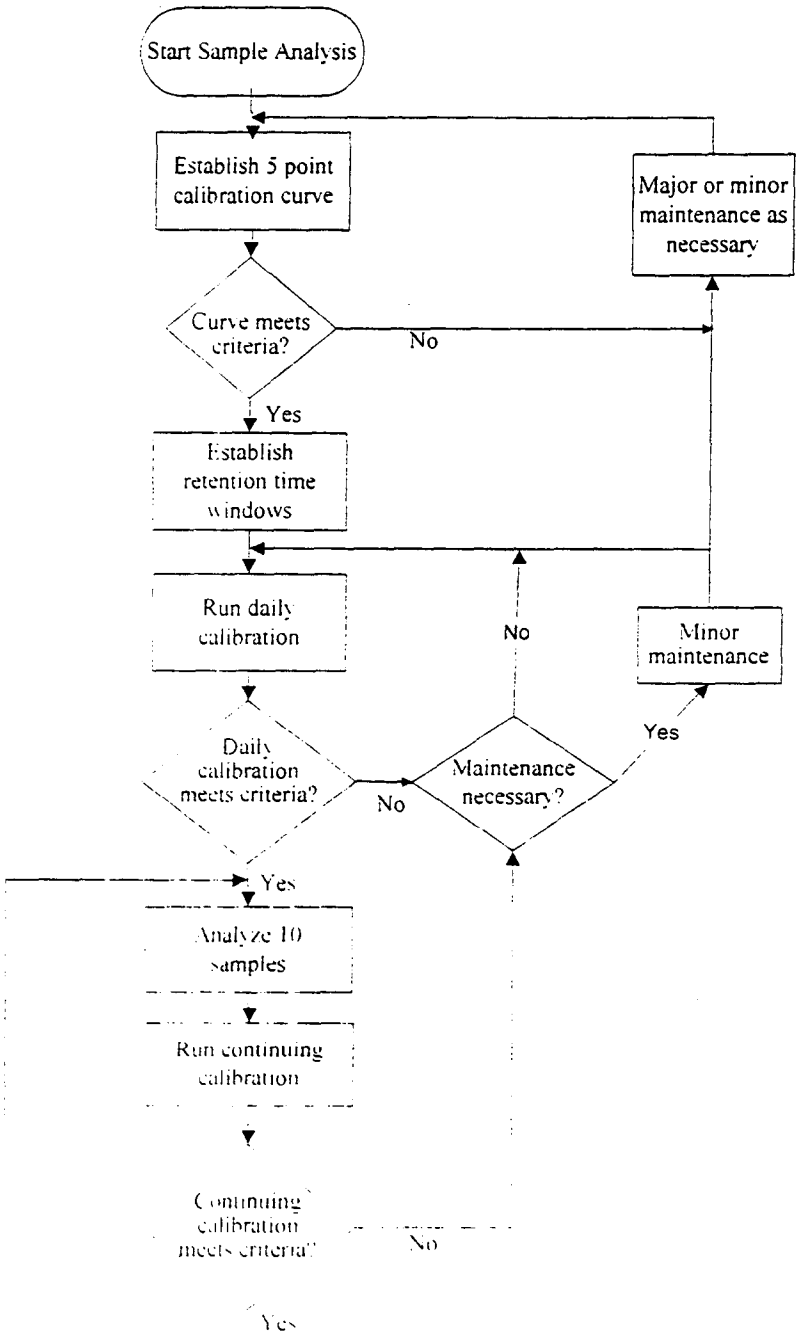
17.4. Flow Diagrams

17.4.1. Initial demonstration and MDL[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt

17.4.2. Sample Analysis[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure. All requirements of the 8000A section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are soluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature (40°C required for low level soils), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a Photoionization Detector (PID, Method 8020A), an Electrolytic Conductivity Detector (ELCD, Method 8010B), or a combination of both (Method 8021A).

2.2. For soil samples with a high level of contamination, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this SOP.

4. INTERFERENCES

4.1. Refer to section 4 of the method 8000A part of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. When utilizing an autosampler system which has multiple ports for sample analysis, it is likely that only a single stage or port may be contaminated by a highly concentrated sample. If a port is suspect, a water blank should be analyzed to verify lack of contamination. If the water blank and subsequent blanks on that port show contamination consistent with the concentrated sample, further maintenance is required. This may include replacing or cleaning the multi-port valve, transfer lines, etc.

4.6. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 14 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 14 day period prior to the analysis of the contaminated holding blank is required.

4.7. Acidification of samples may result in hydrolysis of 2-chloroethyl vinyl ether.

5. SAFETY

5.1. Refer to section 5 of the Method 8000A section of this SOP for general safety requirements.

5.2. Often, purge vessels on purge-and-trap instrumentation are pressurized by the time analysis is completed. Therefore, vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.3. The toxicity or carcinogenicity of each chemical used in this procedure has not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Methanol -- Flammable and toxic

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes -- 10uL, 25uL, 100uL, 250uL, 500uL, and 1000uL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.

6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.

6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.7. Columns - Refer to Table A-2 for details of columns.

6.8. Volumetric flasks, Class A: 5 mL to 250 mL

6.9. pH paper

6.10. Balance capable of weighing to 0.01g for samples.

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000A section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. Suggested methods for generating organic free water include:

- Filtration through a carbon bed.
- Continuously sparging water with helium or nitrogen.
- Use of commercial water purification systems.

Other methods may be used, so long as the requirement that the water not show any interferences is met. The procedure used should be documented in a lab specific attachment.

7.3. Methanol -- Purge and Trap Grade

7.4. Standards

Refer to tables A-5, A-6, A-7 and A-8 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.4.1. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.2. Solid samples are not preserved.

8.3. All samples are stored in glass containers with Teflon lined septa at 4°C - - 2°C, with minimum headspace.

8.4. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

8.5. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. The lack of preservation should be addressed in the case narrative.

8.6. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

9. QUALITY CONTROL

9.1. Refer to the method 8000A section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the method 8000A section of this SOP, section 10, for general calibration procedures. The %RSD limits for the initial calibration are given in Tables A-9 and A-10.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For method 8021A, a single column is used and the PID and ELCD detectors are connected in series. This configuration may also be used for simultaneous 8010B/8020A determination.

10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the 8000A section of this SOP for details of initial calibration criteria.

10.3.2. Soil samples must be purged at 40°C, therefore the calibration curve must also be purged at 40°C. A separate calibration, purged at ambient temperature, must be used for aqueous samples. The aqueous calibration may be used for medium level soils.

10.3.3. The low level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Continuing Calibration

10.4.1. Refer to Tables A-9 and A-10 for details of continuing calibration acceptance criteria.

10.4.2. The level 3 calibration standard is used for the continuing calibration.

11. PROCEDURE

11.1. Refer to the method 8000A section of this SOP for general procedural requirements.

11.2. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.2.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.2.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.2.3. After every 10 samples (including LCS, method blank, and MS samples) a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest for the volatile methods.

It is not necessary but it is permissible to update retention time windows with continuing calibrations.

11.3. Confirmation

The PID and ELCD detectors are sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation should be decided in consultation with the client. For method 8021A confirmatory information can be gained by comparing the relative response from the two detectors.

11.4. Aqueous Sample Analysis (Purge and Trap units using sparge vessels)

11.4.1. Depending on the sensitivity of the instrument and capabilities of the purge and trap device, 5, 10, 20, or 25 mL sample volumes may be analyzed. A 5 mL sample volume is recommended.

11.4.2. Rinse a 5 mL (or 25 mL for larger sample volumes) gas-tight syringe with organic free water. Fill the syringe with the sample to be analyzed, and compress to volume.

11.4.3. Check and document the pH of the sample remaining in the VOA vial after loading the syringe.

11.4.4. Spike with the appropriate volume of surrogate/internal standard solution and matrix spike solution (if required) through the barrel of the syringe. Refer to Tables A-5, A-6, A-7 and A-8 for volumes and concentrations of spiking solutions.

11.4.5. Load onto the purge and trap device and start the run.

11.4.6. If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample has a high response for a compound, analysis should be followed by an organic free water blank. It is recognized that during automated unattended analysis, this may not occur. If any potential carryover hits are present in samples following highly contaminated samples, the sample must be reanalyzed to determine if any of these hits are a result of carryover or are actually present in the sample.

11.4.7. Dilutions may be made in gas tight syringes unless the volume of sample used is less than 5 uL, in which case dilution in volumetric flasks will be necessary.

11.4.7.1. Spike with the same volume of surrogate/internal standard solution as used for undiluted samples prior to loading onto the purge and trap device.

11.4.7.2. For Matrix spike / matrix spike duplicates where the sample requires dilution, the sample is spiked after the dilution is performed.

11.5. Low-Level Solids Analysis

This method is based on purging a heated sediment/soil sample mixed with organic free water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.5.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.5.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than *0.5* g. If the sample is contaminated with analytes such that a purge amount less than *0.5* g is appropriate, use the medium level method described in section 11.*6*.

11.5.3. Connect the purge vessel to the purge and trap device.

11.5.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.

11.5.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.5.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.5.7. Soil samples that have low IS recovery when analyzed should be reanalyzed once to confirm matrix effect.

11.6. Medium-level Soil Analysis: This method is based on extracting the soils with methanol. A waste sample is either extracted or diluted, depending on the solubility with methanol. An aliquot of the extract is added to organic free water containing internal standards as appropriate. These extracts are analyzed under the same conditions as aqueous samples.

11.6.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.6.2. Weigh 4 g (wet weight) of sample into a tared 20 mL vial. Note and record the actual weight to 0.1 g.

11.6.3. Quickly add 9.5 mL of methanol and 0.5 mL of surrogate solution. For matrix spikes or LCS, add 9.0 mL of methanol, 0.5 mL of surrogate solution, and 0.5 mL of matrix spiking solution. For method blanks, use 9.5 mL of methanol spiked with 0.5 mL of surrogate solution. Cap the vial.

11.6.4. Shake the vial for 2 min.

11.6.5. Pipet approximately 1.0 mL of the extract into a 1 or 2 mL screwtop autosampler vial for storage, using a disposable pipet. The remainder may be discarded.

11.6.6. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract to the syringe. Add internal standard if used. Load the sample onto the purge and trap device. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

Refer to section 12 of the 8000A section of this SOP.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000A section of this SOP are presented in Tables A9 and A-10. The spiking level should be 20 µg/L.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Edition, Final Update II, September 1994. Sections 8010B, 8020A and 8021A.

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Purge volumes greater than 5 mL (up to 25 mL) may be used as required to meet reporting limits.

17.1.2. The shutoff valve for the sample-measuring syringe has been omitted.

17.1.3. The use of a soil aliquot between 1 g and 5 g is allowed if performance criteria are met.

17.1.4. The method has been extended to include the use of VOA vial sampling purge and trap autosamplers.

17.2. Modifications from previous revision

17.2.1. Recommended internal and surrogate standards have been changed.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.3.1. Refer to the SOP change form on file in North Canton's Quality Assurance office.

17.4. Tables

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
8010B	Bromodichloromethane	75-27-4	1.0	1.0	120
	Bromoform	75-25-2	1.0	1.0	120
	Bromomethane	74-83-9	1.0	1.0	120
	Carbon Tetrachloride	56-23-5	1.0	1.0	120
	Chlorobenzene	108-90-7	1.0	1.0	120
	Chloroethane	70-00-3	1.0	1.0	120
	2-Chloroethyl vinyl ether	110-75-8	5.0	5.0	620
	Chloroform	67-66-3	1.0	1.0	120
	Chloromethane	74-87-3	1.0	1.0	120
	Dibromochloromethane	124-48-1	1.0	1.0	120
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	120
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	120
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	120
	Dichlorodifluoromethane	75-71-8	1.0	1.0	120
	1,1-Dichloroethane	75-34-3	1.0	1.0	120
	1,2-Dichloroethane	107-06-2	1.0	1.0	120
	1,1-Dichloroethene	75-45-4	1.0	1.0	120
	cis-1,2 Dichloroethene	156-59-4	1.0	1.0	120
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	120
	Dichloromethane(DCM)	75-09-2	5.0	5.0	620
	1,2-Dichloropropane	78-87-5	1.0	1.0	120
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	120
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	120
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	120
	Tetrachloroethene	127-18-4	1.0	1.0	120
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	120
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	120
	Trichloroethene	79-01-6	1.0	1.0	120
	Trichlorofluoromethane	75-69-4	1.0	1.0	120
	Vinyl Chloride	75-01-4	1.0	1.0	120
Additional	Benzyl Chloride	100-44-7	5.0	5.0	620

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
analytes for 8010B	Bromobenzene	108-86-1	1.0	1.0	120
	Dibromomethane	74-95-3	1.0	1.0	120
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	120
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	120
8020A	Benzene	71-43-2	1.0	1.0	120
	Chlorobenzene	108-90-7	1.0	1.0	120
	1,2-Dichlorobenzene	75-34-3	1.0	1.0	120
	1,3-Dichlorobenzene	107-06-2	1.0	1.0	120
	1,4-Dichlorobenzene	75-45-4	1.0	1.0	120
	Ethyl Benzene	100-41-4	1.0	1.0	120
	Toluene	108-88-3	1.0	1.0	120
	Xylenes (total)	1330-20-7	1.0	1.0	120
Additional 8020A	1,2,4 Trimethylbenzene	95-63-6	1.0	1.0	120
	1,3,5 Trimethylbenzene	108-67-8	1.0	1.0	120
	Acetone	67-64-1	10	10	1200
	MEK (2-butanone)	78-93-3	5.0	5.0	620
	MIBK (4-methyl-2-pentanone)	108-10-1	5.0	5.0	620
	Naphthalene	91-20-3	2.0	2.0	250
	Styrene	100-42-5	1.0	1.0	120
	Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	1.0	120
8021A	Benzene	71-43-2	1.0	1.0	120
	Bromobenzene	108-86-1	1.0	1.0	120
	Bromochloromethane	74-97-5	1.0	1.0	120
	Bromodichloromethane	75-27-4	1.0	1.0	120
	Bromoform	75-25-2	1.0	1.0	120
	Bromomethane	74-83-9	1.0	1.0	120
	n-butylbenzene	104-51-8	1.0	1.0	120
	sec-Butylbenzene	135-98-8	1.0	1.0	120
	tert-Butylbenzene	98-06-6	1.0	1.0	120
	Carbon Tetrachloride	56-23-5	1.0	1.0	120
	Chlorobenzene	108-90-7	1.0	1.0	120
	Chlorodibromomethane	124-48-1	1.0	1.0	120
	Chloroethane	75-00-3	1.0	1.0	120
	Chloroform	67-66-3	1.0	1.0	120
	Chloromethane	74-87-3	1.0	1.0	120
	2-Chlorotoluene	95-49-8	1.0	1.0	120

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
	4-Chlorotoluene	106-43-4	1.0	1.0	120
	1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0	1.0	120
	1,2-Dibromoethane(EDB)	106-93-4	1.0	1.0	120
	Dibromomethane	74-95-3	1.0	1.0	120
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	120
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	120
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	120
	Dichlorodifluoromethane	75-71-8	1.0	1.0	120
	1,1-Dichloroethane	75-34-3	1.0	1.0	120
	1,2-Dichloroethane	107-06-2	1.0	1.0	120
	1,1-Dichloroethene	75-35-4	1.0	1.0	120
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	120
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	120
	1,2-Dichloropropane	78-87-5	1.0	1.0	120
	1,3-Dichloropropane	142-28-9	1.0	1.0	120
	2,2-Dichloropropane	590-20-7	1.0	1.0	120
	1,1-Dichloropropene	563-58-6	1.0	1.0	120
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	120
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	120
	Ethylbenzene	100-41-4	1.0	1.0	120
	Hexachlorobutadiene	87-68-3	1.0	1.0	120
	Isopropylbenzene	98-82-8	1.0	1.0	120
	p-Isopropyltoluene	99-87-6	1.0	1.0	120
	Methylene Chloride	75-09-2	5.0	5.0	620
	Naphthalene	91-20-3	2.0	2.0	250
	n-Propylbenzene	10306501	1.0	1.0	120
	Styrene	100-42-5	1.0	1.0	120
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	120
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	120
	Tetrachloroethene	127-18-4	1.0	1.0	120
	Toluene	108-88-3	1.0	1.0	120
	1,2,3-Trichlorobenzene	87-61-6	1.0	1.0	120
	1,2,4-Trichlorobenzene	120-82-1	1.0	1.0	120
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	120
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	120

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, ug/L or ug/kg		
			Aqueous	Low Soil	Medium Soil
	Trichloroethene	79-01-6	1.0	1.0	120
	Trichlorofluoromethane	75-69-4	1.0	1.0	120
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	120
	1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	120
	1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	120
	Vinyl Chloride	75-01-4	1.0	1.0	120
	Xylenes (total)	1330-20-7	1.0	1.0	120

The analytes listed as Additional 8010B and Additional 8020A are amenable to these tests and may be analyzed at client request.

Table A-2

Recommended Conditions for Method 8020A

Parameter	Recommended Conditions
Temperature program	50°C, 1min, 10°C/min to 200°C,1min
Column 1	Rtx-502.2 or DB-502.2 60m x 0.53mm 3.0um
Column 2	Rtx-1 or DB-1 60m x 0.53mm 3.0 um
Carrier gas	Helium or hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3

Recommended Conditions for Method 8010B or Method 8010B/8020A

Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-4

Recommended Conditions for Method 8021A

Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-5
Surrogate and Internal Standard Concentrations for Aqueous and Low Level Soil Samples

Standard	Components	Working Solution ug/mL	Spike amount uL (for 5 mL purge)	Final concentration ug/L (ug/kg)
8020A IS/SS	4-Chlorotoluene (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40		40
8010B IS/SS	4-chlorotoluene (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40		40
8021A IS/SS	Fluorobenzene (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40	10	40

It may be necessary to select different surrogates in order to minimize sample interferences. !-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometines be requested as a target analyte for method 8010. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane: Elutes very close to chloroform and cis-1,2-dichloroethene on the 502.2 column. Target analyte for 8021.

1,2-Bromochloroethane:

1-Chloro-2-fluorobenzene: Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2

a,a,a-Trifluorotoluene: Good for 8020, coelutes or very close to trichloroethene

Bromofluorobenzene: Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.

2-Bromo-1-chloropropane: May coelute with 1,1,2-trichloroethane

Table A-6

Surrogate and Internal Standard Concentrations for Medium Level Soil Samples

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Conc in 4g sample µg/kg	Conc in purged extract µg/L (100µL/5 mL)
8020A SS	4-chlorotoluene (SS)	20	500	2500	20
8020A IS	1-chloro-4-fluorobenzene	20	5	N/A	20
8010B SS	4-chlorotoluene (SS)	20	500	2500	20
8010B IS	1-chloro-4-fluorobenzene	20	5	N/A	20
8021A SS	Fluorobenzene	20	500	2500	20
	1-4-Dichlorobutane	20	500	2500	20
8021A IS	1-chloro-4-fluorobenzene	20	5	N/A	20

Table A-7

Concentrations for LCS and MS/MSD compounds, low level soil and aqueous

Standard	Components	Working Solution ug/mL	Spike amount uL (5 mL purge)	Final concentration ug/L (ug/kg)
8020A MS	Benzene	10	5	10
	Toluene	10		10
	Chlorobenzene	10		10
8010B MS	Chlorobenzene	10	5	10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10
8021A MS	Benzene	10	5	10
	Toluene	10		10
	Chlorobenzene	10		10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10

Table A-8

Concentrations for LCS and MS/MSD compounds, medium level soil

Standard	Components	Working Solution ug/mL	Spike amount uL	Final concentration ug/L (ug/kg)
8020A MS	Benzene	10	500	10
	Toluene	10		10
	Chlorobenzene	10		10
8010B MS	Chlorobenzene	10	500	10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10
8021A MS	Benzene	10	500	10
	Toluene	10		10
	Chlorobenzene	10		10
	1,1-Dichloroethene	10		10
	Trichloroethene	10		10

Table A-9

Continuing Calibration and Initial Demonstration Limits

Analyte	Continuing Calibration and Initial Demonstration Recovery Limits, +/- %	Initial Demonstration RSD Limits, %
Method 8010B		
Bromodichloromethane	24.0	21.5
Bromoform	26.5	23.5
Bromomethane	41.5	38
Carbon Tetrachloride	31.5	28
Chlorobenzene	28.0	25
Chloroethane	23.0	22
2-Chloroethyl vinyl ether	40.0	41.5
Chloroform	25.0	22.5
Chloromethane	40.5	37
Dibromochloromethane	34.5	31.5
1,2-Dichlorobenzene	30.0	27.5
1,3-Dichlorobenzene	50.5	45.5
1,4-Dichlorobenzene	30.5	27.5
1,1-Dichloroethane	16.0	16
1,2-Dichloroethane	28.5	26
1,1-Dichloroethene	37.0	33
trans-1,2-Dichloroethene	36.0	32
Dichloromethane	22.5	20
1,2-Dichloropropane	26.0	26
cis-1,3-Dichloropropene	36.0	36.5
trans-1,3-Dichloropropene	36.0	36.5
1,1,2,2-Tetrachloroethane	51.0	46
Tetrachloroethene	30.0	27
1,1,1-Trichloroethane	29.0	24.5
1,1,2-Trichloroethane	21.5	19.5
Trichloroethene	23.0	21
Trichlorofluoromethane	33.5	30
Vinyl chloride	31.5	28.5
Additional compounds	35	25

Table A-10		
Continuing Calibration and Initial Demonstration Limits		
Analyte	Continuing Calibration and Initial Demonstration Recovery Limits, +/- %	Initial Demonstration RSD Limits, %
Method 8020A		
Benzene	23.0	20.5
Chlorobenzene	19.5	17.5
1,2-Dichlorobenzene	32.0	29.0
1,3-Dichlorobenzene	27.5	25.0
1,4-Dichlorobenzene	30.5	27.5
Ethylbenzene	37.0	33.5
Toluene	22.5	20.0
Additional compounds	35.0	25.0

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8000A is applied to the analysis of organochlorine pesticides and Aroclors (PCBs) by GC/ECD. This Appendix is to be applied when SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate Quanterra sample extraction SOPs. (CORP-OP-0001)

Table B-1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000A section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001.

APPENDIX C

LABORATORY ANALYSIS CRITERIA

Table 3
Summary of Method Quality Objectives for Method 8021
VOCS

QC Element	**Target Analyte / Surrogate**	Poor Purgers / Gases / **Sporadic Marginal Failures'**
Initial Calibration (9.2.2.1)	Primary Evaluation: r ≥ 0.995, RSD ≤ 20%, r^2 ≥ 0.990	No allowance
ICV (9.3)	1. Recovery = 85 -115% 2. **QC/MRL**: D ≤ 15%	Sporadic Marginal Failures': Recovery = 70 - 130%
CCV (9.5 / 9.5.2 9.5.2.1)	1. Primary Evaluation: Drift ≤ 15%, D ≤ 15% Alternative Evaluation: Mean Drift/D For all target analytes ≤ 15% 2. **QC/MRL**: D ≤ 15%	Primary Evaluation: Drift ≤ 20%, D ≤ 20% Alternative Evaluation: Maximum allowable Drift/D For each target analyte ≤ 30%
MB (10.2.1 / 11.4.1)	Target Analytes: Analytes < MDL Check Sample (-2X MDL)	Common Lab Contaminants: Analytes < MRL
LCS (10.2.2 / 11.4.2)	Water : Recovery, 80 - 120% Soil: Recovery, 75 – 125%	Sporadic Marginal Failures': Recovery = 60 - 140%
IVIS (10.2.3/ 11.4.3/ 11.4.3.2)	Recovery = 70 - 130%	Sporadic Marginal Failures': %Rec = 60% - 140%
MSD/MD (10.2.4 / 11.4.4)	Water: RPD ≤ 30 Soil: No RPD Limits	Water: RPD ≤ 40 Soil: No RPD Limits
Surrogates (10.2.5 11.4.5)	Interference-Free Matrix: Water: Recovery 80 - 120% Soil: Recovery 75 - 125% Project Sample Matrix: Recovery = 70 - 130%	Not Applicable
Target Analyte Confirmation (12.3)	RPD ≤ 40	RPD ≤ 40

[1] The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if the 8020 Target Analyte List (10 compounds) is reported, 1 SMF is allowed. If the 8010 Target Analyte List (32 compounds) is reported, 3 SMFs are allowed. Ifthefull8021 Target Analyte List (60 compounds) is reported, 4 SMFs are allowed. If the MS includes only a subset of compounds, allow only one (1) SMF for that QC element. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

APPENDIX D

DATA VALIDATION CHECKLIST

VOLITILE ORGANIC DATA VALIDATION CHECKLIST

Laboratory _____

SDG Number _____

Section I-Part A is filled out if the data package contains VOA analyses.

The review consisted of checking and verifying that the following performance criteria are within acceptable QC limits.

- x Holding Time Review
- x System Monitoring Compound (Surrogate) Review
- x Matrix Spike/Matrix Spike Duplicate Review
- x Blank Contamination Review
- x GC/MS Instrument Performance Check
- x Initial and Continuing Calibration Check Internal Standard Areas Check

Any exceedance in the QC limits are documented in the attached summary sheets. Please note that field/rinse blanks, holding times for non-detects, as well as qualifying detected values below the CRQL will be analyzed as a separate process.

The following qualifiers may be applied:
J = Positive result at an estimated value
R = Data is unreliable due to significant QC problems
B = Blank contamination

Data Reviewer _____

VOA ANLYSES

1. Chain-of-Custody Records and Requests for Analyses (C-O-C/RFA)

Are C-O-C/RFA Records present for all samples?
[ ] Yes [ ] No [ ] N/A

Do the C-O-C/Request for Analysis or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems, or special circumstances affecting the quality of the data? [ ] Yes [ ] No [ ] N/A

Action: Use professional judgment to evaluate the effect on the quality of the data.

Note: Holding times information will be available from the IRDMIS database. Only verification will be performed during validation.

2. System Monitoring Compound (Surrogates! Analysis

Are all system monitoring compound recoveries below the upper acceptance limit (IJL)?
[ ] Yes [ ] No [ ] N/A

Action: If a system monitoring compound recovery is above the upper acceptance level (UL) qualify all associated positive values as estimated "J".

Are all system monitoring compound recoveries above the lower acceptance level (LL)?
[ ] Yes [ ] No [ ] N/A

Action: If a system monitoring compound recovery is below the lower acceptance limit (LL) qualify all associated detected data estimated "J".

Note: Professional judgment should be used to qualify data that have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard areas.

Common Lab Contaminants - Methylene Chloride, Acetone, Toluene, 2-butanone, Carbon Disulfide

Note: Trip blanks are used to qualify only those samples with which they were shipped and are only required for VOA matrices. Blanks may not be qualified because of contamination in another blank. Field Blanks and Trip Blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems. Field blank contamination is not to be applied at this time. This will be done as a separate step in the validation process. If all associated samples for the Trip Blanks are not found in the package please note the exceptions on the "TRIP BLANK TO BE VALIDATED LIST". Do not apply qualifiers to rinse blanks, (which are denoted as R~SW under Site Type on the COC Form).

Has an instrument performance compound been analyzed for every 12 hours of sample analysis per instrument. [ ] Yes [ ] No [ ] N/A

Action: Reject (R) all data generated outside an acceptable 12-hour tune interval.

Have the ion abundance's been normalized to m/z - 9j?
[ ] Yes [ ] No [ ] N/A

Action: If mass assignment is in error, qualify all associated data as unusable (R).

7. GC/IV~S Initial Calibration

Are the Initial Calibration Forms present and complete for the volatile fraction?
[ ] Yes [ ] No [ ] N/A

Action: If any calibration standard forms are missing, please contact lab.

Are the RRFs above 0.05 for Target/HSL compounds?
[ ] Yes [ ] No [ ] N/A

Action: If the RRF is below the above listed criteria qualify all associated analyze detected values estimated "J" and rejects "R" all associated analyze non-detects.

Are any % RSD's above 30% for any sample analyze?

[ ] Yes [ ] No [ ] N/A

Action: If %RSD >30% qualify all associated detected sample analyses estimated "J".

8. Continuing Calibration

Are all continuing calibration forms present and complete for the volatile fraction?

[ ] Yes [ ] No [ ] N/A

Action: If any continuing calibration forms are missing or incomplete, please notify laboratory.

Are all RRFs above 0.05 for Target/HCL compounds?

[ ] Yes [ ] No [ ] N/A

Action: If the RRF is below the above mentioned criteria qualify all detected values as estimated "J" and all non-detects as rejected "R".

Are any % D's above 25% for any volatile analyze?

[ ] Yes [ ] No [ ] N/A

Action: If any %D exceeds 25% qualify all associated detected sample analyses as estimated "J"

9. Internal Standard

Are the internal standard areas of every sample and blank within the upper and lower limits for each continuing calibration?

[ ] Yes [ ] No [ ] N/A

Action: If the internal standard area count is outside the upper or lower limit, flag "J" all associated positive results. If extremely low area counts (~ 25%) are reported, or if performance exhibits a major abrupt drop-off, qualify all associated non-detects as "R", rejected and all associated detects as estimated, "J".

Note: If the IS area is above 150%, use professional judgment.

Are the retention times of the internal standards within 30 seconds of the associated calibration standard? [ ] Yes [ ] No [ ] N/A

Action: Professional judgment should be used to qualify data if the retention times differ by more than 30 seconds to determine if a-false positive or negative exists.

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: SW846 Method 8021 Standard List

Matrix: SOLID
Extraction: PURGE AND TRAP - 5 mL purge
Method: Volatile Organics (8021B)
QC Program: STANDARD TEST SET
Location: Quanterra - North Canton

Target List 2313		Detection Limits				Check List 914					Spike List 915				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Benzene	1	ug/kg	0.27	ug/kg	19980303			69	132	20			62	150	33
Bromobenzene	1	ug/kg	0.45	ug/kg	19980303										
Bromochloromethane	1	ug/kg	0.17	ug/kg	19980303										
Bromodichloromethane	1	ug/kg	0.23	ug/kg	19980303										
Bromoform	1	ug/kg	0.35	ug/kg	19980303										
Bromomethane	1	ug/kg	0.16	ug/kg	19980303										
n-Butylbenzene	1	ug/kg	0.45	ug/kg	19980303										
sec-Butylbenzene	1	ug/kg	0.47	ug/kg	19980303										
tert-Butylbenzene	1	ug/kg	0.47	ug/kg	19980303										
Carbon tetrachloride	1	ug/kg	0.4	ug/kg	19980303										
Chlorobenzene	1	ug/kg	0.65	ug/kg	19970609			51	131	20			10	142	32
Chlorodibromomethane	1	ug/kg	0.32	ug/kg	19980303										
Chloroethane	1	ug/kg	0.15	ug/kg	19980303										
Chloroform	1	ug/kg	0.26	ug/kg	19980303										
Chloromethane	1	ug/kg	0.15	ug/kg	19980303										
2-Chlorotoluene	1	ug/kg	0.41	ug/kg	19980303										
4-Chlorotoluene	1	ug/kg	0.52	ug/kg	19980303										
1,2-Dibromo-3-chloropropane (DBCP)	1	ug/kg			0										
1,2-Dibromoethane (EDB)	1	ug/kg	0.24	ug/kg	19980303										
Dibromomethane	1	ug/kg	0.24	ug/kg	19980303										
1,2-Dichlorobenzene	1	ug/kg	0.69	ug/kg	19970609										
1,3-Dichlorobenzene	1	ug/kg	0.78	ug/kg	19970609										
1,4-Dichlorobenzene	1	ug/kg	0.76	ug/kg	19970609										
Dichlorodifluoromethane	1	ug/kg	0.18	ug/kg	19980303										
1,1-Dichloroethane	1	ug/kg	0.18	ug/kg	19980303										
1,2-Dichloroethane	1	ug/kg	0.21	ug/kg	19980303										
cis-1,2-Dichloroethene	1	ug/kg	0.67	ug/kg	19980303										
trans-1,2-Dichloroethene	1	ug/kg	0.21	ug/kg	19980303										
1,1-Dichloroethene	1	ug/kg	0.82	ug/kg	19970609			29	140	20			10	203	38
1,2-Dichloropropane	1	ug/kg	0.2	ug/kg	19980303										
1,3-Dichloropropane	1	ug/kg	0.17	ug/kg	19980303										
2,2-Dichloropropane	1	ug/kg	0.45	ug/kg	19980303										
cis-1,3-Dichloropropene	1	ug/kg	0.55	ug/kg	19980303										
trans-1,3-Dichloropropene	1	ug/kg	0.54	ug/kg	19980303										
1,1-Dichloropropene	1	ug/kg	0.4	ug/kg	19980303										
Ethylbenzene	1	ug/kg	0.46	ug/kg	19980303										

Printed at /99 11:19:14 AM

Target Analyte List: Q: SW846 Method 8021 Standard List

Matrix: SOLID
Extraction: PURGE AND TRAP - 5 mL purge
Method: Volatile Organics (8021B)
QC Program: STANDARD TEST SET
Location: Quanterra - North Canton

Target List 2313		Detection Limits				Check List 914					Spike List 915				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Hexachlorobutadiene	1	ug/kg	0.48	ug/kg	19980303										
Isopropylbenzene	1	ug/kg	0.56	ug/kg	19980303										
p-Isopropyltoluene	1	ug/kg	0.96	ug/kg	19980303										
Methylene chloride	5	ug/kg	0.15	ug/kg	19980303										
Naphthalene	1	ug/kg	0.25	ug/kg	19980303										
n-Propylbenzene	1	ug/kg	0.43	ug/kg	19980303										
Styrene	1	ug/kg			0										
1,1,1,2-Tetrachloroethane	1	ug/kg	0.18	ug/kg	19980303										
1,1,2,2-Tetrachloroethane	1	ug/kg	0.88	ug/kg	19980303										
Tetrachloroethene	1	ug/kg			0										
Toluene	1	ug/kg	0.41	ug/kg	19980303			66	129	20			50	142	40
1,2,3-Trichlorobenzene	1	ug/kg	0.88	ug/kg	19980303										
1,2,4-Trichlorobenzene	1	ug/kg	0.44	ug/kg	19980303										
1,1,1-Trichloroethane	1	ug/kg	0.22	ug/kg	19980303										
1,1,2-Trichloroethane	1	ug/kg	0.31	ug/kg	19980303										
Trichloroethene	1	ug/kg	0.64	ug/kg	19970609			10	216	20			10	216	45
Trichlorofluoromethane	1	ug/kg	0.17	ug/kg	19980303										
1,2,3-Trichloropropane	1	ug/kg			0										
1,2,4-Trimethylbenzene	1	ug/kg	0.47	ug/kg	19980303										
1,3,5-Trimethylbenzene	1	ug/kg			0										
Vinyl chloride	1	ug/kg	0.14	ug/kg	19980303										
Xylenes (total)	1	ug/kg			0										
1,4-Dichlorobutane								50	150	0			50	150	0
Trifluorotoluene								50	150	0			50	150	0

Quanterra Reference Data Summary

Target Analyte List: Q: SW846 Method 8021 Standard List

Matrix: WATER
Extraction: PURGE AND TRAP - 5 mL purge
Method: Volatile Organics (8021B)
QC Program: STANDARD TEST SET
Location: Quanterra - North Canton

Target List 2313		Detection Limits				Check List 914					Spike List 915				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Benzene	1	ug/L	0.013	ug/L	19980219			73	134	20			55	161	25
Bromobenzene	1	ug/L	0.18	ug/L	19980225										
Bromochloromethane	1	ug/L	0.23	ug/L	19980225										
Bromodichloromethane	1	ug/L	0.56	ug/L	19980218										
Bromoform	1	ug/L	0.4	ug/L	19980218										
Bromomethane	1	ug/L	0.29	ug/L	19980225										
n-Butylbenzene	1	ug/L			0										
sec-Butylbenzene	1	ug/L			0										
tert-Butylbenzene	1	ug/L			0										
Carbon tetrachloride	1	ug/L	0.59	ug/L	19980218										
Chlorobenzene	1	ug/L	0.51	ug/L	19980218			61	134	20			42	147	23
Chlorodibromomethane	1	ug/L	0.34	ug/L	19980218										
Chloroethane	1	ug/L	0.5	ug/L	19980218										
Chloroform	1	ug/L	0.37	ug/L	19980218										
Chloromethane	1	ug/L	0.4	ug/L	19980218										
2-Chlorotoluene	1	ug/L	0.39	ug/L	19980218										
4-Chlorotoluene	1	ug/L	0.4	ug/L	19980218										
1,2-Dibromo-3-chloropropane (DBCP)	1	ug/L	0.6	ug/L	19980218										
1,2-Dibromoethane (EDB)	1	ug/L	0.43	ug/L	19980218										
Dibromomethane	1	ug/L	0.42	ug/L	19980218										
1,2-Dichlorobenzene	1	ug/L	0.38	ug/L	19980218										
1,3-Dichlorobenzene	1	ug/L	0.26	ug/L	19980218										
1,4-Dichlorobenzene	1	ug/L	0.45	ug/L	19980218										
Dichlorodifluoromethane	1	ug/L	0.26	ug/L	19980218										
1,1-Dichloroethane	1	ug/L	0.38	ug/L	19980218										
1,2-Dichloroethane	1	ug/L	0.37	ug/L	19980218										
cis-1,2-Dichloroethene	1	ug/L	0.78	ug/L	19980218										
trans-1,2-Dichloroethene	1	ug/L	0.41	ug/L	19980218										
1,1-Dichloroethene	1	ug/L	0.27	ug/L	19980218			35	127	20			14	151	28
1,2-Dichloropropane	1	ug/L	0.52	ug/L	19980218										
1,3-Dichloropropane	1	ug/L	0.18	ug/L	19980225										
2,2-Dichloropropane	1	ug/L	0.78	ug/L	19980218										
cis-1,3-Dichloropropene	1	ug/L	0.6	ug/L	19980313										
trans-1,3-Dichloropropene	1	ug/L	0.32	ug/L	19980218										
1,1-Dichloropropene	1	ug/L	0.59	ug/L	19980218										
Ethylbenzene	1	ug/L	0.026	ug/L	19980219										

Target Analyte List: Q: SW846 Method 8021 Standard List

Matrix: WATER
Extraction: PURGE AND TRAP - 5 mL purge
Method: Volatile Organics (8021B)
QC Program: STANDARD TEST SET
Location: Quanterra - North Canton

Target List 2313		Detection Limits				Check List 914					Spike List 915				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Hexachlorobutadiene	1	ug/L	0.25	ug/L	19980225										
Isopropylbenzene	1	ug/L			0										
p-Isopropyltoluene	1	ug/L			0										
Methylene chloride	5	ug/L	0.36	ug/L	19980218										
Naphthalene	1	ug/L	0.54	ug/L	19980219										
n-Propylbenzene	1	ug/L			0										
Styrene	1	ug/L			0										
1,1,1,2-Tetrachloroethane	1	ug/L	0.65	ug/L	19980218										
1,1,2,2-Tetrachloroethane	1	ug/L	0.32	ug/L	19980225										
Tetrachloroethene	1	ug/L	0.2	ug/L	19980225										
Toluene	1	ug/L	0.024	ug/L	19980219			71	132	20			55	159	25
1,2,3-Trichlorobenzene	1	ug/L	0.21	ug/L	19980218										
1,2,4-Trichlorobenzene	1	ug/L	0.57	ug/L	19980218										
1,1,1-Trichloroethane	1	ug/L	0.37	ug/L	19980218										
1,1,2-Trichloroethane	1	ug/L	0.4	ug/L	19980218										
Trichloroethene	1	ug/L	0.41	ug/L	19980218			58	131	20			10	229	41
Trichlorofluoromethane	1	ug/L	0.39	ug/L	19980218										
1,2,3-Trichloropropane	1	ug/L	0.73	ug/L	19980218										
1,2,4-Trimethylbenzene	1	ug/L	0.054	ug/L	19980219										
1,3,5-Trimethylbenzene	1	ug/L	0.04	ug/L	19980219										
Vinyl chloride	1	ug/L	0.82	ug/L	19980218										
Xylenes (total)	1	ug/L	0.3	ug/L	19990427										
1,4-Dichlorobutane								50	150	0			50	150	0
Trifluorotoluene								50	150	0			50	150	0

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.

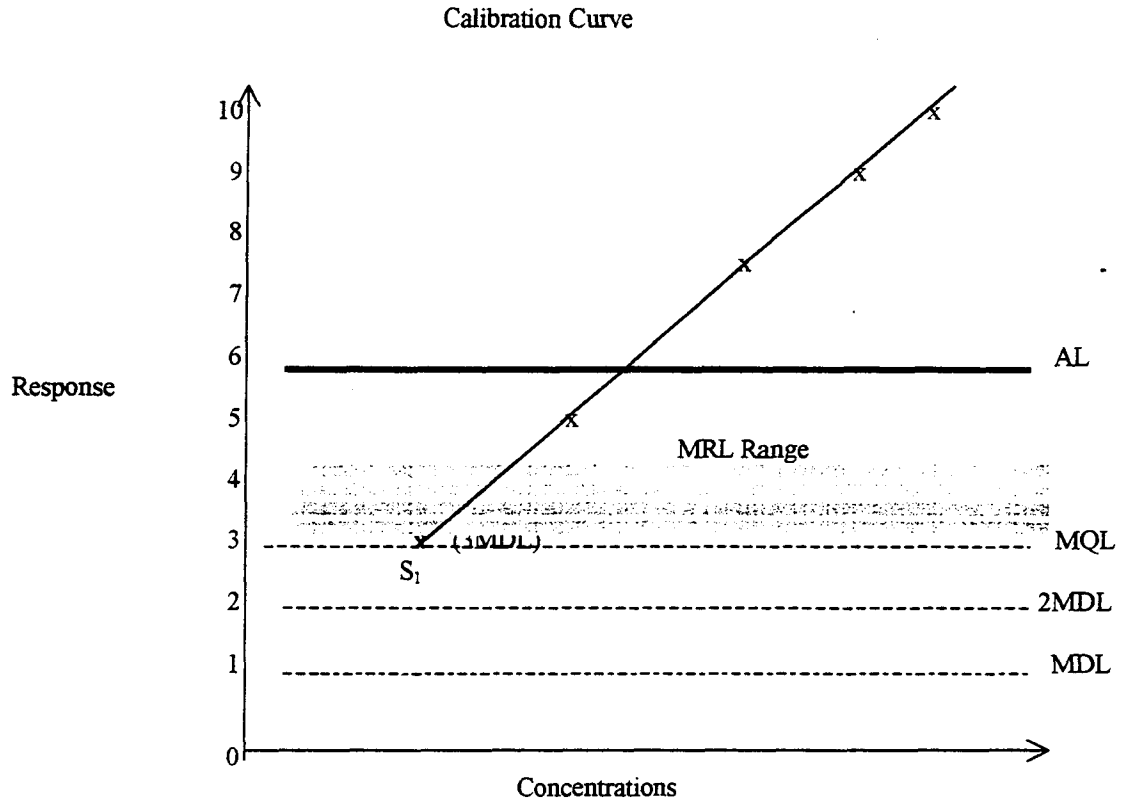
4. MRL: Method Reporting Limits are threshold values above which results are reported as positive quantities, and below which results are reported as non-detect (<) and flagged with "U", or as estimated and flagged with "J". MRL must not be established at levels below the MDL check sample level. MRL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

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.

2. General Comment: The flow of the document is very confusing as it jumps back and forth between the entire Plum Brook Ordnance Works (PBOW) history and the history of the Lower Toluene Tank Area. Please create a Plum Brook Ordnance Works general history section and focus the remainder of the document on the area to be investigated.

Response: The report will be edited accordingly.

3. Section 1.2.1 Facility Size and Borders: Please indicate the size and borders of the Lower Toluene Tank Area.

Response: The Lower Toluene Tank Area is approximately 4 acres. It is located in the southwestern portion of PBOW. The Tank Area is bordered to the south by the intersection of Patrol road and Taylor Road. The area is bordered to the east by abandoned railroad tracks running north and south. The remaining borders to the north and west are thick vegetative and forested areas. This information will be added to section 1.2.1.

4. Section 1.3 Past Data Collection Activities: Please Provide boring logs for the referenced wells as attached figures.

Response: The referenced boring logs will be added to the figure section of the QAPP.

5. Section 1.5.1 Project Target parameters and Intended Data Usages: The Target Parameters for this investigation should include analysis for Lead and Semivolatile Organic Compounds (SVOCs). Historically, Department of defense (DOD) facilities

utilized lead based paint for painting above ground storage tanks. SVOCs from the rail spur may have impacted the area as well. The Field Sampling Plan and Data Quality Objectives sections of this document should be revised to include these parameters as well.

Response: The reviewer's point is valid and will be taken into consideration for any future work performed at the site. The primary focus for this limited site investigation was to determine if the toluene stored within the tanks had adversely impacted the site through spills and leaks. The sampling outlined in the Field Sampling Plan and DQOs has already been completed. This comment will be addressed further in the Final Report responses.

U.S. ARMY CORPS
OF ENGINEERS
LOUISVILLE DISTRICT

Field Sampling Plan

Limited Site Investigation of the former

Plum Brook Ordnance Works

Lower Toluene Tank Area

Sandusky, Ohio

DERP-FUDS HTRW Project No. G05OH001824

July 2000

TABLE OF CONTENTS

1.0 PROJECT DESCRIPTION 1

1.1 Introduction 1

1.2 History and Usage 1

1.2.1 Installation 1
1.2.2 Site Specific 2

1.3 Climate 3

1.4 Topography. 3

1.5 Geology 3

1.6 Hydrogeology 4

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES 4

2.1 General. 4

2.2 Planning Team Members. 5

2.3 Louisville Technical Team. 5

3.0 SCOPE AND OBJECTIVES 5

4.0 SAMPLING DESIGN AND RATIONALE 6

5.0 FIELD ACTIVITIES AND SAMPLING PROCEDURES 7

5.1 Visual Inspections 7

5.2 Sample Documentation 7

5.3 Photographs 8

5.4 Surface and Subsurface Soil Sampling 8

5.5 Surface Water and Sediment Sampling 9

5.6 Field Quality Control Sampling 9

5.7 Decontamination 10

6.0 FIELD SAMPLE IDENTIFICATION AND CUSTODY PROCEDURES 11

6.1 Sample Containers, Preservation and Holding Times 12

6.2 Sample Identification 13

6.3 Sample Packaging 14

6.4 Custody Transfer and Shipment Procedures 15

7.0 DISPOSITION OF FIELD INVESTIGATION DERIVED WASTE (IDW) 15

8.0 SCHEDULE 15

8.1 Start Date 15

8.2 Pre-mobilization 15

8.3 Mobilization 16

8.4 Demobilization 16

Attachment:

Headspace Field Screening - PID

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 installation by several studies has been related to past activities and is being addressed by the DOD under the Defense Environmental Restoration Program (DERP), Formerly Used Defense Sites (FUDS). A limited Site Investigation (SI) of the "Lower Toluene Storage Tanks" will be conducted to evaluate the potential for contamination of the former tank area that may have resulted from past DOD activities.

This project is one of several limited SI projects simultaneously undertaken by the US 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

During the period that PBOW was operational, DoD constructed and utilized six storage tanks that served as bulk storage for toluene used in the production of TNT. PBOW had three TNT production areas A, B, and C. Each production area was supported by two aboveground storage tanks (AST) used for bulk toluene storage. Each tank was approximately 30 feet in diameter, 41 feet high, constructed of steel, and had a capacity of 200,000 gallons. Each tank rested on a concrete foundation surrounded by a 1-foot wide by 1-foot high containment berm. The berm emptied into a 3-foot square drain valve pit. The pump for each tank is located at ground level near the drain valve pit. Historical photographs show much larger earthern berms around each tank which were approximately 55 feet from the center of the tank. More recent historical photographs do not show an earthern berm around the tanks. It has not been ascertained if these berms were removed by DoD, NASA, or naturally eroded away. The "Lower Toluene Tanks" supported "TNT Area C" during PBOW operations. The tanks were numbered 645 (northern tank) and 655 (southern tank). The tanks were installed in 1941, and were in service until 1945and are illustrated in Figure 1. Toluene was transported to and from the tank area by railroad. The tanks were decommissioned in 1945 by pumping out their contents, draining transfer lines, and opening top and bottom flanges for ventilation. NASA

removed tank 655 from the lower tank area and installed it near the reactor facility.

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 it's 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 the lower toluene storage tank area is located primarily on a flat grassland area at an elevation of approximately 680 feet mean sea level (msl). The tank area is located north of the Taylor Road and Patrol Road intersection, and is southeast of "TNT Area C". Mature trees and vegetation surround the remaining tank. There are no buildings within the area. Some abandoned single-track railroad lines are located to the east of the tank area. Precipitation runoff from the tank area accumulates in a nearby surface depression or pond west of the site, or flows northward to a tributary of Pipe Creek by means of a drainage ditch. The surface depression was identified in an Inventory Project Request (INPR) prepared by CELRH (USACE, 1998) for the lower toluene tank area, however it could not be clearly identified from historical or recent aerial photographs.

1.5 Geology

It is assumed that two formations of the Devonian Age underlay the lower toluene tank area. The lowermost and oldest formation is the Delaware Limestone. It is characterized as a hard, dense, finely crystalline limestone and dolomite. Dissolution of this unit has been described which has produced solution channels along bedding planes and joints, and even producing caverns in some areas. The unit is typically buff colored and usually is described as 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 tank area. 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 tank area at an approximate depth of 10 feet below ground surface (IT, 1999).

The overburden at the tank area is predominately composed of glacial till, outwash, or lacustrine (lake) deposits. The overburden at the lower tank area is approximately 10 feet thick (IT, 1999). Morrison Knudsen installed two monitoring wells, MK-MW-14 (installed north of removed Tank 655) and MK-MW-15 (installed north of Tank 645). The boring logs of these two wells described the lithology of the overburden unit as brown silty sand with traces of clay and some organics, until bedrock is encountered at depths of 11.5 feet (MK-MW-14) and 9 feet (MK-MW-15).

1.6 Hydrogeology

Based on a review of the site-wide groundwater study (IT, 1999), ground water is encountered in the overburden soils underlying the tank area. Groundwater in the overburden soils is generally expected to be purged or trapped water related to the permeable interface between the overburden and underlying bedrock. The groundwater encountered at this site generally occurs in the granular zones within the soil overburden. These sources of water contain widely varying quantities of water depending on recent precipitation. Based on expected soil types, overburden groundwater quantities, if encountered, should be minimal and unrelated to deeper potable ground water sources within the bedrock layer.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 General.

This work is being pursued by the United States Army Corps of Engineers (USACE) under 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 - Kieth Peecook
Project Manager: Rick Meadows, CELRH-DL-M
Project Engineer: 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
Toxicologist: David Brancato, CELRL-ED-EE
Hydrogeologist: Martin Wahking, CELRL-ED-EB
QA/QC Chemist: Samir Mansy, CELRL-ED-EB
Technical Manager: 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 analyses will be compared to risk based, media-specific screening criteria. United States Environmental Protection Agency (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

The target parameter is toluene. This parameter will be identified based on analytical results using USEPA SW-846 methodology. The analytical 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

Toluene is the only constituent of concern (COC) associated with the tank area that may have been released to the environment. The population to be represented by sampling in this investigation is the range of concentrations of toluene that may exist in the surface water, surface and subsurface soil within the toluene tank area. Total Organic Carbon (TOC) analysis, pH, and geotechnical data will be collected on soil samples to aid in assessing the potential for migration through the soil column at the site. The potential release of contamination, the chemical properties of the suspected COC, the geological conditions, and potential transport mechanisms support the recommended sampling strategy.

Because different populations of COC may exist in different soil strata, more than one stratum or matrix will be characterized by sampling. 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 soil overburden. Surface water samples will be collected to evaluate potential COC migration pathways associated with drainage. The following is the rationale for the proposed location and number of samples:

- A visual survey will be performed at the lower toluene tank area to locate the concrete berm of removed tank 655, the surface depression to the west of the area, visual signs of contamination, and additional seeps or drainage patterns.
- Four hand-augered borings will be drilled approximately 40 feet to the east, west, south, and north of each tank location. These borings will be within the original earthen berm depicted in historical photographs. Soil samples will be collected from the first foot of each boring. The remaining depths will be screened, at 1-foot intervals to a final depth of 4 feet, with a photoionization detector (PID). An additional 4-5 samples within these borings will be collected based on PID screening. Based on the most recent ground water results toluene was not present at either well in the lower toluene tank area. Any residual toluene associated with the tanks should be found within the first 4 feet of soil.
- Additional hand-augered borings will be drilled within close proximity of the pump and drain valves and associated pipes leading to the tanks. One boring will be located at the pump, and two will be located at the midpoint of the piping between the pump and both tanks. One sample will be collected from the first foot of soil at the pump. The two remaining borings will be screened at 1-foot intervals with the PID to a total depth of 2 feet. The decision to collect samples from these borings will be made in the field based on screening results. These areas were selected because of associated leaks that might have occurred during tank loading and unloading, and possible leaks along the piping.

- If it can be located, one surface water and sediment sample will be collected from the surface depression located to the west of the tank area. If the depression is located but is not holding water a boring will be centrally located within the depression and screened. The decision to collect a sample will made in the field based on screening results. This area was selected to determine if the COC has migrated away from the tanks via the depression.

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.

- Decontamination of sampling equipment.
- Disposition of 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 to identify each picture taken. Prints will be identified with the project number, date and time taken, and a brief description of the subject, location, and orientation of the photograph.

5.4 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 will press the auger into the ground. Sampling will be continuous throughout the boring. Samples will be collected in 1-foot increments down to 4 feet. All borings will be documented with a hand auger boring log and soils will be 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. The analytical samples will be placed immediately in the appropriate container for BTEX analysis and quickly sealed. All soil sample containers will be clearly identified and preserved on ice at 4° Celsius. A portion of the sample will be placed in a Ziploc Freezer bag for headspace field screening with a Photo ionization detector (PID). The PID will be used to screen for toluene. The sample will be allowed to equilibrate with the headspace within the bag for 15 minutes. After the allotted time a PID measurement will be recorded by placing the tip of the

PID into the bag. A reading will be taken and documented in the field log book. All samples collected within the first foot of each boring will go to the laboratory for BTEX analyses. A maximum of 5 additional samples will be sent to the laboratory for BTEX analysis based on the highest field screening data. Any variances in sampling due to field observations and conditions will be properly recorded in the field logbook. Samples will also be collected for geotechnical, and TOC analyses. The geotechnical samples will confirm the visual soil classifications made in the field and will provide information about the physical properties of the soil.

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. Sample collection information will be recorded in a 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. Any variances in sampling due to field conditions or findings will be properly documented in the field logbook.

5.5 Surface Water and Sediment Sampling

Each pair of surface water and sediment samples 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 will be collected facing upstream. Sediment samples will be collected by using a stainless steel scoop attached to a pole and collecting the sediment. All samples will be collected and quickly sealed so that loss of volatiles is minimal. All samples will be clearly and preserved on ice 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 samples will be analyzed for BTEX.

- Field Blanks - Field blanks will be collected from the deionized water used in the field for sampling procedures. One field blank will be collected for every 20 investigative samples. These samples will monitor field-sampling procedures for the introduction of contamination. Field blanks will be analyzed for both BTEX analyses.

- 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.

- Trip Blanks - These samples will be supplied by the laboratory. They will consist of deionized water. These samples will be placed in all coolers containing BTEX samples and will monitor for contamination 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 equipment and sampling equipment. Several gallons of clean, distilled water will be maintained on site along with plastic buckets, brushes, soap, etc., to decontaminate personnel and 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) generated as a result of decontamination procedures will be labeled and stored until final disposal arrangements, consistent with applicable environmental requirements, are made.

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. Rinsates and decontamination fluids will require containerization approved for liquids, labeled and properly stored, while awaiting approval for 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 (see figure). 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 table 1 and 2. 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 1 - Sample Containers, Preservation Methods, and Holding Times for Water Samples

Parameter	Analytical Method	Quantity	Type	Preservation Methods	Holding Times
BTEX	8021	2	40 ml, amber glass vials, Teflon®-lined cap	Cool, 4°C	Analysis: 7 days

Table 2 - Sample Containers, Preservation Methods, and Holding Times for Soils Samples

Parameter	Analytical Method	Quantity	Type	Preservation Methods	Holding Times
BTEX	8021	1	4-ounce, wide-mouth, amber glass, Teflon®-lined cap	Cool, 4°C	Analysis: 14 days
TOC & Geotechnical	Walkly-Black	1	4-ounce, wide-mouth, amber glass, Teflon®-lined cap	Cool, 4°C	28 Days

6.2 Sample Identification

Each sample will be assigned a unique identification number 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 T1 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 project. For this project the code is:

T1 - Lower toluene tank

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

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 via priority overnight service by the courier. This will allow for the least amount of time from sampling to analysis. The Field Team Leader will perform notification of sample shipment to the laboratory.

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 field activities. 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.

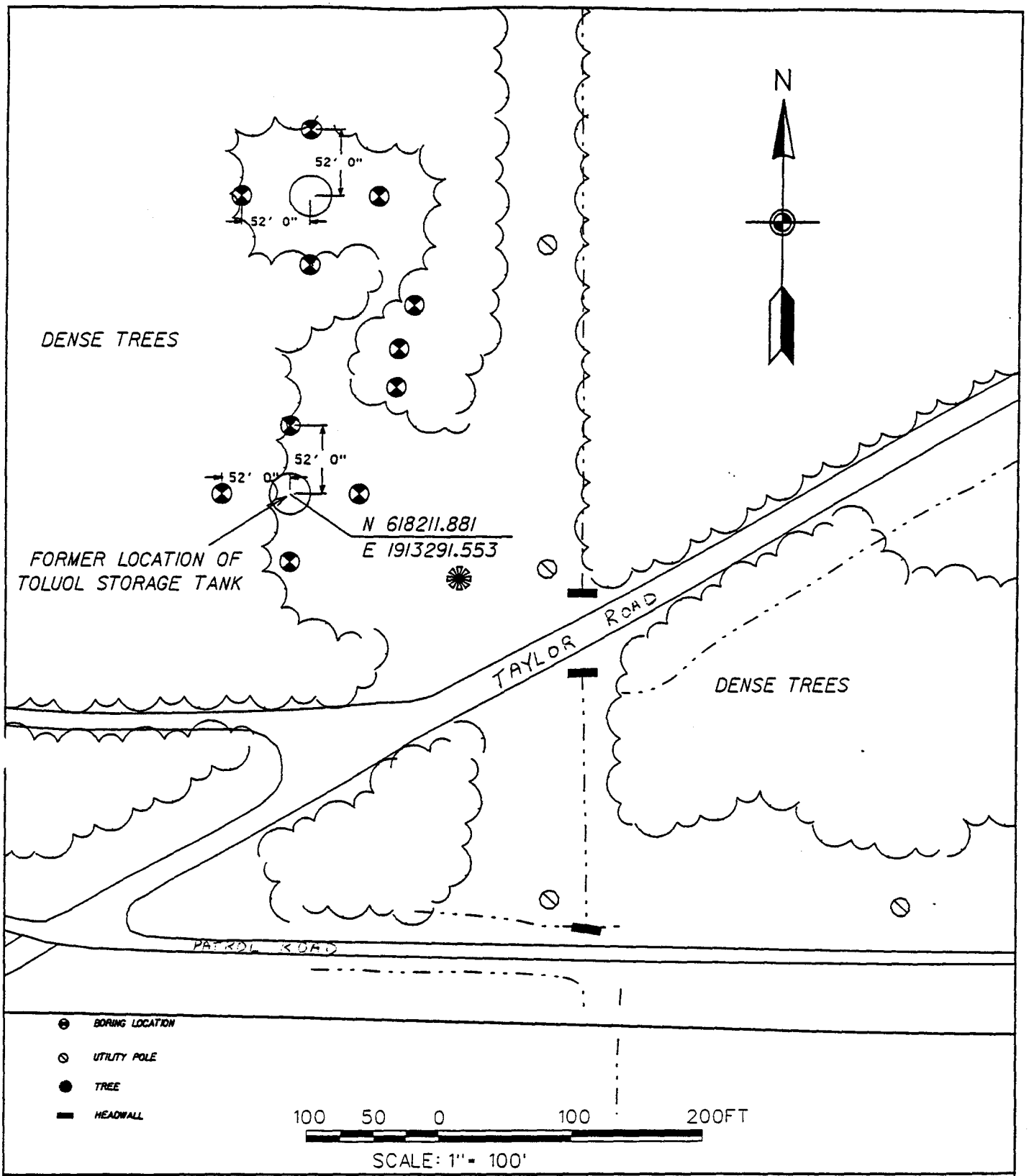
- Site Visit - A site visit will take place prior to any sampling event. This site visit will allow USACE personnel to visually assess sites, and will facilitate optimization of the sampling design and rationale.

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 FSP and QAPP prepared for the site investigation. All sampling equipment and materials will be inspected for proper decontamination and good working condition. All provisions will be made by USACE to ensure that field supplies are available and appropriate for sampling team members. These supplies include logbooks, sample containers, labels, chain of custody forms, shipping supplies, coolers, and packing materials.

8.4 Demobilization

At the completion of sampling activities USACE personnel will demobilize. Arrangements have been made for the disposal of investigation-derived waste (IDW). 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.



Design By:	Drawn By:	Checked By:	US Army Corps of Engineers	PLUM BROOK STATION SANDUSKY, OHIO LIMITED SITE INVESTIGATION FY 99	PROPOSED SAMPLE LOCATIONS LOWER TOLUENE TANKS
Reviewed By:	Approved By:				
Date: 10 MAY 99	Scale: 1" = 100'	Drawing Code:	Sheet Ref. No.	Sht 3 of 7	

File Name:
Plot Date:

Attachment

Headspace Field Screening Procedures at Lower Toluene Tanks

1.1 Equipment List

The following list of equipment will be needed to headspace analysis of soil samples:

- Ziploc Freezer Bags
- Photovac model 2020, Organic vapor analyzer equipped with a photoionization detector (PID), 100 ppm isobutylene gas cyliner for field calibration check.
- Field book
- Waterproof and permanent marking pens

1.2 Field Screening Procedures

A portion of each soil sample collected for headspace analysis will be placed in the appropriate labeled bag. The container will be filled with approximately 4-8 ounces of sample matrix. The bag will be sealed and the sample matrix will be allowed to equilibrate with the headspace for 15 minutes. Care will be taken in the selection of soils with respect to consistency and sample placement in the container in order to achieve comparability and consistency.

The sample headspace in the bag will be analyzed with the photoionization analyzer by slightly opening the bag at the corner (about one half of an inch) and inserting the instrument probe through the bag. Care must be taken in the insertion of the probe as not to compromise the integrity of the seal. If the seal has been compromised, this will be recorded appropriately or a new sample taken if possible.

1.3 Photoionization Detector (PID)

The selection of a PID as the appropriate organic vapor analyzer will be based on contaminants of concern and/or ambient conditions at the respective site. It is anticipated that a PID detector will be used for all the work.

1.4 Calibration

The instrument(s) selected for use in accordance with data quality objectives and site requirements will be calibrated according to the manufacturer recommendations and specifications. Daily calibration checks will be performed by connecting the PID to a small gas cylinder containing 100 ppm isobutylene. Once the cylinder valve is opened a reading will be taken and recorded in the field log book. The acceptance criteria for the calibration check will be 85 - 115 ppm readings. If this criteria is not met the PID will be re-calibrated per the manufacturer recommendations and specifications. Calibration checks will be performed at the start of each field work day, and every 8 hours there after.

2.5 *Documentation*

All procedures and field conditions will be recorded on the boring log and in the field logbook. The record will include a description of the material being screened as well as site conditions such as humidity and equilibration time and temperature.